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AFWAL-TR-87-2042
Volume XIV

PRODUCTION OF JET FUELS FROM COAL-DERIVED LIQUIDS

VOL XIV - Oxygenates Content of Coal-Derived Jet Fuels

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JUNE 1990

INTERIM REPORT FOR THE PERIOD 26 NOVEMBER 1986 - 31 JULY 1989

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This technical report has been reviewed and is approved for publication.

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION: Unclassified	1b. RESTRICTIVE MARKINGS: None
2a. SECURITY CLASSIFICATION AUTHORITY: N/A	3. DISTRIBUTION/AVAILABILITY OF REPORT: Approved for public release; Distribution is unlimited
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE: N/A	
4. PERFORMING ORGANIZATION REPORT NUMBER(S): N/A	5. MONITORING ORGANIZATION REPORT NUMBER(S): AFWAL-TR-87-2042 Vol XIV
6a. NAME OF PERFORMING ORGANIZATION: University of North Dakota Energy & Environmental Research Cntr; 6b. OFFICE SYMBOL (If applicable):	7a. NAME OF MONITORING ORGANIZATION (WRDC/POSF): Aero Propulsion and Power Laboratory Wright Research and Development Center
6c. ADDRESS (City, State, and ZIP Code): Grand Forks ND 58202	7b. ADDRESS (City, State, and ZIP Code): Wright-Patterson AFB OH 45463-6563
8a. NAME OF FUNDING/SPONSORING ORGANIZATION: N/A; 8b. OFFICE SYMBOL (If applicable): N/A	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: FY1455-86-N0657
8c. ADDRESS (City, State, and ZIP Code): N/A	10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
63216F	2480	16	01

11. TITLE (Include Security Classification)
Production of Jet Fuels from Coal-Derived Liquid, Vol XIV - Oxygenates Content of Coal-Derived Jet Fuels

12. PERSONAL AUTHOR(S)
C. L. Knudson

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Interim	FROM 861126 TO 890731	June 1990	63

16. SUPPLEMENTARY NOTATION
None

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	Jet Fuels, Coal-Derived Jet Fuels; Coal, Coal Liquids; JP-4, JP-8, JP-8X, Oxygenates Fuel Properties; Fuel Composition, Stability

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Samples of jet fuel (JP-4, JP-8, JP-8X) produced from the liquid by-products of the gasification of lignite coal from the Great Plains Gasification Plant were analyzed to determine the quantity and type of organo-oxygen compounds present. Results were compared to similar fuel samples produced from petroleum. Large quantities of oxygen compounds were found in the coal-derived liquids and were removed in the refining process. Trace quantities of organo-oxygenate compounds were suspected to be present in the refined fuels. Compounds were identified and quantified as part of an effort to determine the effect of these compounds in fuel instability. Results of the analysis showed trace levels of phenols, naphthols, benzofurans, hexanol, and hydrogenated naphthols were present in levels below 100 ppm.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT: ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION: Unclassified
22a. NAME OF RESPONSIBLE INDIVIDUAL: William E. Harrison III	22b. TELEPHONE (Include Area Code): 513-255-6601; 22c. OFFICE SYMBOL: WRDC/POSF

DD Form 1473, JUN 86 — *Previous editions are obsolete.* — SECURITY CLASSIFICATION OF THIS PAGE: UNCLASSIFIED

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FOREWORD

In September 1986, the Fuels Branch of the Aero Propulsion and Power Laboratory at Wright-Patterson Air Force Base, Ohio, commenced an investigation of the potential for production of jet fuel from the liquid by-product streams produced by the gasification of lignite at the Great Plains Gasification Plant in Beulah, North Dakota. Funding was provided to the Department of Energy (DOE) Pittsburgh Energy Technology Center (PETC) to administer the experimental portion of this effort. This report details the effort of the University of North Dakota Energy and Environmental Research Center (UNDEERC), who, as a contractor of DOE (DOE Contract No. DE-AC22-87PC90016), characterized these liquid by-product streams. DOE/PETC was funded through Military Interdepartmental Purchase Request (MIPR) FY1455-86-N0657. Mr. William E. Harrison III was the Air Force project engineer, Mr. Gary Stiegel was the DOE/PETC project engineer, and Dr. Warrack Willson was the UNDEERC project manager.

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EXECUTIVE SUMMARY

As part of the "Production of Jet Fuels From Coal-Derived Liquids" project, three coal-derived jet fuels from small-scale processing tests and one coal-derived bulk jet fuel from a large-scale test were produced by Amoco Research Center. Samples from these tests were provided to the University of North Dakota Energy and Environmental Research Center (EERC) and the Western Research Institute (WRI) with a request to determine the oxygenates present in the samples. In addition, samples of petroleum-derived jet fuels were obtained for comparative purposes. Some compositional and physical data were obtained during the study. The following summarizes the results of the work.

<u>Petroleum JP-4 and JP-8</u>

- No oxygenates present.
- Appear to be simple distillation cuts, unblended.
- Primarily composed of a series of normal and branched alkanes with some BTX present.

<u>Coal-derived Jet Fuels</u>.

- JP-4 is primarily cyclohexanes with some toluene and xylene.
- JP-8 is primarily more highly alkylated cyclohexanes and decalins and is a blend of different distillate cuts.
- JP-8X is primarily decalins.
- Bulk JP-8 is essentially the same as the initial JP-8 sample.
- Only phenolics in the 1-3 ppm range were detected in the bulk sample.
- Olefins of less than 0.2 wt% were observed.
- Petroleum-derived jet fuel contained primarily straight-chained and branched aliphatics whereas the coal-derived (for these processing methods) contained mainly hydrogenated aromatics (cyclohexanes and decalins).

Gas chromatography/mass spectrometry (GC/MS) and selected ion mode (SIM) for data acquisition was used to determine oxygenates in the fuels. The concentration of species was determined using internal standards. In a related study, WRI determined that of 21 phenolics, for which sensitivity factors were determined, all were present in concentrations less than the 100 ppm detection level. EERC analyzed for four phenols, two naphthols, two benzofurans, hexanol, and hydrogenated naphthol using two methods of sample preparation. Using the method that had a detection limit of 10 ppm, only a nonquantifiable, trace amount of phenolics was detected. Using an extraction method, detectability was decreased to 1 ppm. At this detection level, the bulk sample indicated the presence of 2 ppm phenol, 3 ppm o-cresol, and 1 ppm 2,4-dimethylphenol. Trace amounts of 2,3,5-trimethylphenol and 2-naphthol were also detected in the bulk sample. No 2-methyl-1-naphthol was detected.

The levels of oxygenates in the coal-derived fuels are in the low ppm range. A total of 6 ppm of phenolics was detected in the bulk sample of coal-derived JP-8.

GOALS

The primary goal of this research was to determine the level of oxygenates present in coal-derived jet fuel produced in small-scale tests and in a large-scale bulk sample.

INTRODUCTION

There were a number of goals and objectives for the jet fuels research effort conducted at the University of North Dakota Energy and Mineral Research Center. These were accomplished with both in-house work and through subcontracts to other researchers. The work has been documented in a series of topical reports, of which this report is the last. The overall project objective was to determine the feasibility of producing different types of jet fuel from coal-derived liquids. The following work was performed and reported (topical report titles indicate the information contained in the report and its objective) during the course of the contract with UNDEMRC.

AFWAL-TR-87-2042, "PRODUCTION OF JET FUELS FROM COAL-DERIVED LIQUIDS"

Volume I - Market Assessment for Liquid By-Products from the Great Plains Gasification Plant, by J.E. Sinor. August 1987.
Subcontract to J.E. Sinor Consultants, Inc., Niwot, Colorado.

Volume II - Characterization of Liquid By-Products from the Great Plains Gasification Plant, by C.L. Knudson and T.A. Aulich. May 1988.

Volume VIII - Heteroatom Removal by Catalytic Processing, by J.R. Rindt, M.D. Hetland, C.L. Knudson, and W.G. Willson. January 1989.

Volume XIV - Oxygenates Content of Coal-Derived Jet Fuels, by C.L. Knudson. June 1990.

During the course of the total research effort a number of samples were characterized in support of various parts of the project.

Sample	Source
Feedstock:	
Rectisol Naphtha Stream	Dakota Gasification Company
Crude Phenol Stream	Dakota Gasification Company
Tar Oil Stream	Dakota Gasification Company
Processing Streams:	
Dynaphen process samples	Hydrocarbon Research, Inc.
Internal process samples	UNDEMRC

Product Streams:

Petroleum-derived

JP-4	Typical jet fuel, WRDC
JP-8	Typical jet fuel, WRDC

Coal-derived, initial processing

JP-4	Amoco Oil Company
JP-8	Amoco Oil Company
JP-8X	Amoco Oil Company

Coal-derived, bulk processing

Bulk JP-8	Amoco Oil Company

The analytical methods used and developed on the feedstocks were reported in Volume II. The Dakota Gasification Company requested more detailed characterization of the rectisol naphtha material in a separate contract. A copy of the final report is attached as Appendix A. The results of the analysis of the Dynaphen process samples were reported in a separate report (attached as Appendix B). Data determined on the internal processing samples produced at UNDEMRC are included in Volume VIII. The work performed on the jet fuel samples at UNDEMRC is included in this report while the work performed at WRI on these same samples is reported as a separate document. The analytical data for feedstocks and jet fuel products from the above reports and this report provide the following insights concerning the samples characterized:

Rectisol Naphtha

- Volatile, primarily BTX material, making it too light for jet fuel.
- Olefins present in each distillation fraction.
- Contains about 50 ppm of pyridine compounds (nitrogen bases).
- Light sulfur compounds produced throughout distillation.
- Over 70% of the total sulfur concentrated in the distillate bottoms (10 wt% of the total sample with a sulfur content of 8 wt%).

Crude Phenol

- Primarily phenol, cresol, and xylenol compounds.
- Contains guaiacols, dihydroxybenzenes (catechols, etc.), and naphthalene in small amounts.
- During the initial distillation, azeotropes with water selectively decrease the amount of contaminants.
- Would hydrotreat to form C6-to-C8 cyclohexanes, which are too light for jet fuel.

Tar oil

- Contains aliphatics (~8 wt%), aromatics (~48%), phenolics (~36%), nitrogen bases (~3%), water (~4%), and trace solids.
- Light phenolics and water can be removed by distillation.
- Solids and heavy ends cannot be removed by simple distillation due to instability related to heating rate.
- Nitrogen bases present are known catalyst poisons and are difficult to hydrotreat.
- Would hydrotreat initially to form tetralins (multiple ring hydrocarbons containing one aromatic ring) that would require additional hydrotreating

to produce decalins and hydrocracking to produce cyclohexanes.

Petroleum JP-4 and JP-8
- Appear to be simple distillation cuts, unblended.
- Primarily composed of a series of normal and branched alkanes with some BTX present.

Coal-derived Jet Fuels
- JP-4 is primarily cyclohexanes with some toluene and xylene.
- JP-8 is primarily more highly alkylated cyclohexanes and decalins and is a blend of different distillate cuts.
- JP-8X is primarily decalins.
- Bulk JP-8 is essentially the same as the initial JP-8 sample.
- Only phenolics in the low ppm range (~5ppm) were observed.
- Petroleum-derived jet fuel contained primarily straight-chained and branched aliphatics, whereas the coal-derived (for these processing methods) contained mainly hydrogenated aromatics (cyclohexanes and decalins).

The following presents the research results on the compositional and oxygenate determinations performed at UNDEERC.

The selection of oxygenates to quantitatively analyze for in the jet fuels was based on the composition of the feed material (tar oil) and potential intermediate products and are listed as follows:

Initial Compounds	Intermediate Products
Phenol	Cyclohexanol
o-Cresol	1-Methylcyclohexanol
2,4-Dimethylphenol	
2,3,4-Trimethylphenol	
2-Naphthol	Decahydro-2-naphthol
2-Methyl-1-naphthol	
Benzofuran	
Dibenzofuran	

EXPERIMENTAL

All quantitative analyses of the trace oxygenates were performed using a Hewlett-Packard Model 5988 GC/MS equipped with a 60-m DB-5 capillary column (250-um i.d., 0.25-um film thickness) identical to that used for GC/FID analysis. Each test fuel was analyzed by two GC/MS methods. Method 1 utilized an injection of 20-nL neat sample of each fuel with a split ratio of ca. 1:50. Selected ion monitoring (SIM) was used to selectively detect each of the oxygenated standards listed in Table 1. Two mass ions that are characteristic of each of the standard oxygenates were monitored at appropriate retention time intervals. Since the petroleum-derived JP-8 sample was found to have no detectable concentrations of the standard oxygenates, it was used to prepare standard concentrations of each of the oxygenates. Standards in petroleum JP-8 were prepared at 100-, 10-, and 1-ppm (wt/wt)concentrations, and each standard solution contained 20 ppm of 1-chloronaphthalene as an internal standard. Quantitative values were based on the presence of a chromatographic peak having the correct mass and the same retention time as the standard oxygenates in the petroleum JP-8. The results of the SIM oxygenates analyses are given in Table 1. In general, any error in these analyses should give an artificially high value (because of the possibility of a co-eluting matrix species having the same mass ions as the target oxygenate).

Because of the extremely complex SIM chromatograms, and in order to decrease the detection limits, the phenolic concentrations were also determined using a second method. Each fuel sample and the standards (in petroleum JP-8) were spiked with 1-ppm d_5-phenol as an internal standard. Ten ml of each sample and standard were then extracted three times with 3 ml of 0.5 N NaOH. The water phase containing the phenolics was then acidified to a pH of 3, and the phenolics were recovered by extracting three times with 3 ml portions of methylene chloride. The methylene chloride extracts were then analyzed using GC/MS (scan range of 50-400 amu) and 1-uL on-column injections. Quantitation was based on the integrated areas (divided by the area of the internal standard) of each oxygenate. Table 2 shows the results of these analyses.

The analytical methods to characterize the jet fuels employed in this work are GC/FID for concentration determinations and GC/MS as well as retention index data for identification. For a mixture of compounds containing primarily carbon and hydrogen, the area% values obtained using an FID detector are within 5% of the wt% values. Therefore, area% and wt% are used interchangeably in this report. Retention index values were determined by analysis of a sample before and after the addition of a known amount of a mixture containing C6-to-C25 normal alkanes. This also provides an identification of the normal alkanes present in a mixture. Analytic methods are presented in detail in Volume II.

RESULTS AND DISCUSSION

Oxygenates Analysis

In general, the results of the two analysis methods give reasonable quantitative agreement. The concentrations of the test oxygenates were present in the fuels at or below detection limits (1 or 10 ppm) in almost every case. Only the bulk JP-8 sample showed any analytically significant concentrations of the oxygenates (phenol, ortho-cresol, and 2,4-dimethylphenol at 2,3, and 1 ppm, respectively).

TABLE 1

ESTIMATED CONCENTRATIONS OF OXYGENATED COMPOUNDS IN JET FUELS[a]

Compound	Detection Limit (ppm, wt/wt)	Petroleum-Derived JP-4	Petroleum-Derived JP-8	Coal-Derived JP-4	Coal-Derived JP-8	Coal-Derived JP-8x	Coal-Derived Bulk
cyclohexanol	10	ND[b]	ND	ND	ND	ND	ND
1-methylcyclohexanol	10	ND	ND	ND	ND	ND	ND
phenol	10	ND	ND	ND	ND	ND	ND
benzofuran	1	ND	ND	ND	ND	ND	trace[c]
o-cresol	1	ND	ND	ND	ND	ND	trace
2,4-dimethylphenol	10	ND	ND	ND	ND	ND	trace
2,3,5-trimethylphenol	10	ND	ND	ND	ND	ND	trace
decahydro-2-naphthol	10	ND	ND	ND	ND	ND	10
2-naphthol	10	ND	ND	ND	ND	ND	ND
dibenzofuran	10	ND	ND	ND	10	10	20
2-methyl-1-naphthol	1	ND	ND	ND	-[d]	-	-

a) Selected ion monitoring GC/MS techniques were used.
b) ND = not detected.
c) Chromatographic peaks having the correct mass and retention times were detected, but their integrated areas were less than those of the standards for the reported detection limits.
d) Could not be determined due to interference from matrix species.

TABLE 2

ESTIMATED CONCENTRATIONS OF OXYGENATED COMPOUNDS IN JET FUELS USING BASE EXTRACTION AND FULL SCAN GC/MS[a]

Compound	Detection Limit (ppm, wt/wt)	Petroleum-Derived JP-4	Petroleum-Derived JP-8	Coal-Derived JP-4	Coal-Derived JP-8	Coal-Derived JP-8x	Coal-Derived Bulk
phenol	1	1	ND	trace	trace	trace	2
o-cresol	1	ND	ND	trace	trace	trace	3
2,4-dimethylphenol	1	ND	ND	ND	ND	ND	1
2,3,5-trimethylphenol	1	ND	ND	ND	ND	ND	trace
2-naphthol	1	ND	ND	ND	ND	trace	trace
2-methyl-1-naphthol	1	ND	ND	ND	trace	2	ND

a) ND = not detected, while trace means that the chromatographic peaks having the correct mass and retention times were detected, but their integrated areas were less than those of the standards for the reported detection limits.

Simulated Distillation and GC/MS Data

Figures 1 to 5 depict the simulated distillation data for petroleum-derived JP-8, interim coal-derived JP-4, JP-8, and JP-8X, and the bulk coal-derived JP-8 samples, respectively. The coal-derived JP-8 contains more lighter boiling compounds than the petroleum-derived JP-8 (Figures 1 and 3). The small-test coal-derived JP-8 sample and the large-scale bulk JP-8 sample have similar distillation profiles (Figures 3 and 5) except that the former has less material in the 400-430°F region (the JP-8X boiling point region). As expected, the simulated distillation data for coal-derived JP-4, JP-8, and JP-8X indicate each represents a higher boiling point cut.

Figure 6 depicts the relationship between the simulated distillation curve and the component data obtained by GC analysis of the bulk coal-derived JP-8 sample. The distillation curve was created by the summation of the individual component peaks present in the GC area% trace. The top and bottom scales on Figure 6 relate the boiling point of normal alkanes to temperature. Figures 7 and 8 depict the GC component traces for the various samples. The vertical scale for these traces is in area% (0.76 wt%/cm). The horizontal scale is indexed against normal alkanes. The major compounds and amounts present in coal-derived JP-4 are listed at the top of Figure 7. Coal-derived JP-4, JP-8, and JP-8X each contain the sample compounds in greater or lesser amounts.

The detailed GC/FID/MS data from the coal-derived jet fuels is presented in Table 3. The following abbreviations were used to simplify naming:

n-C#	Normal or straight-chained alkanes.
C#-	The number of carbon groups attached to the named compound (C1- for methyl, C2- for 2 methyl or one ethyl, etc.).
BCN	Trans-bicyclo(4.0.3)nonane.
TCTD	Tricyclotetradecane.
TCHP	Tetradecahydrophenanthrene.

The coal-derived jet fuels are rich in naphthenes (cyclopropanes, cyclohexanes, and decalins) and contain less of partially hydrogenated aromatics (tetralins, tetradecahydrophenanthrene, etc.) and little aromatics (benzenes). As expected, the cyclohexanes are most prevalent in JP-4, while the decalins and tetralins are most prevalent in the heavier JP-8X.

Petroleum-Derived JP-8

The simulated distillation and GC component data for the petroleum-derived JP-8 is depicted in Figures 1 and 8 (bottom), respectively. There is a strong presence of straight-chained, normal alkanes (C8 to C15) maximized at 10 and 11 carbon atoms in length. The smooth increase and decrease in the concentration of the alkanes indicate that they represent a distillation cut. Various alkylated single-ring aromatics are present in maximum concentrations of 1 wt% each. Most of the background peaks are due to the presence of branched alkanes.

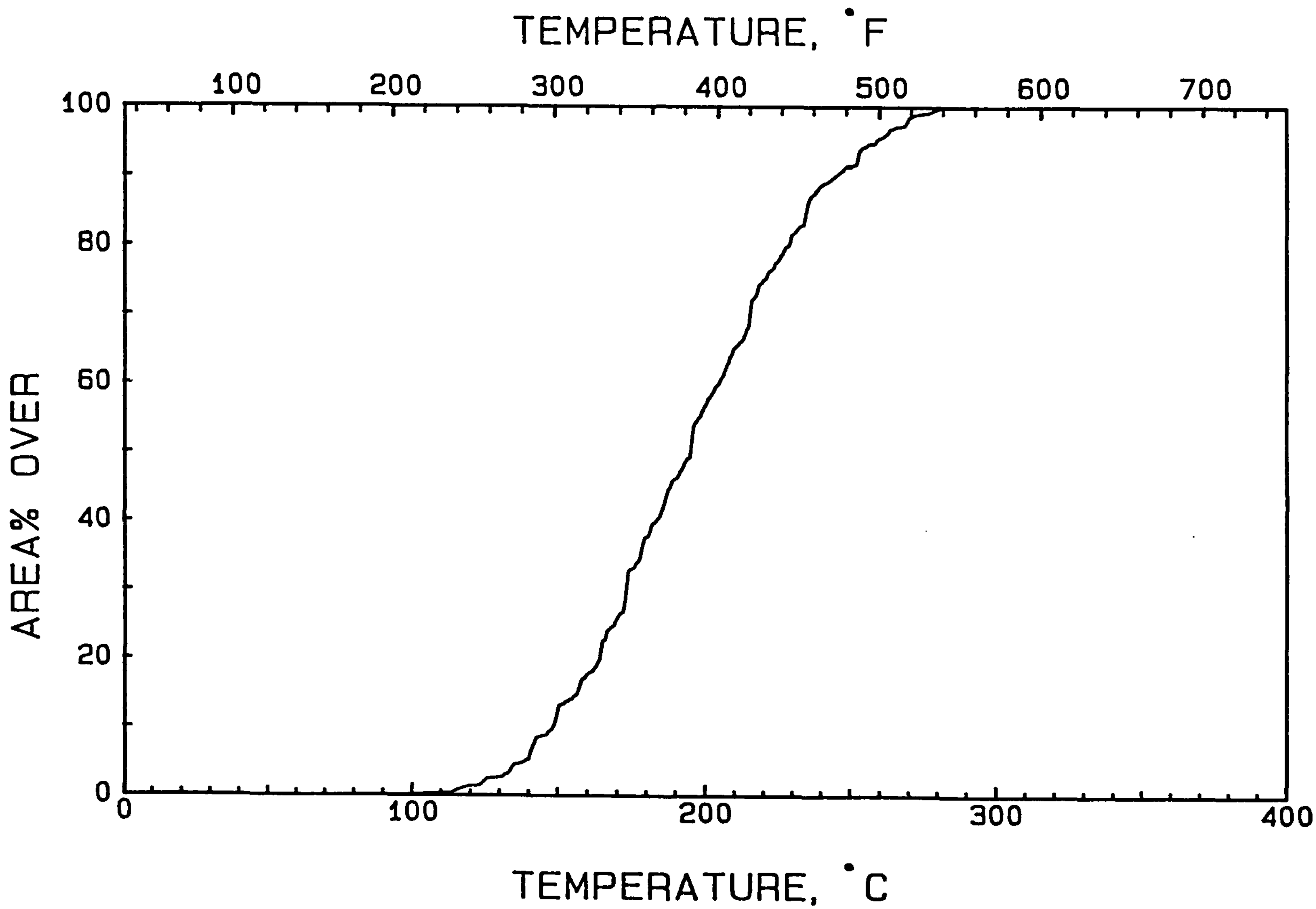

Figure 1. Simulated distillation data for petroleum-derived JP-8.

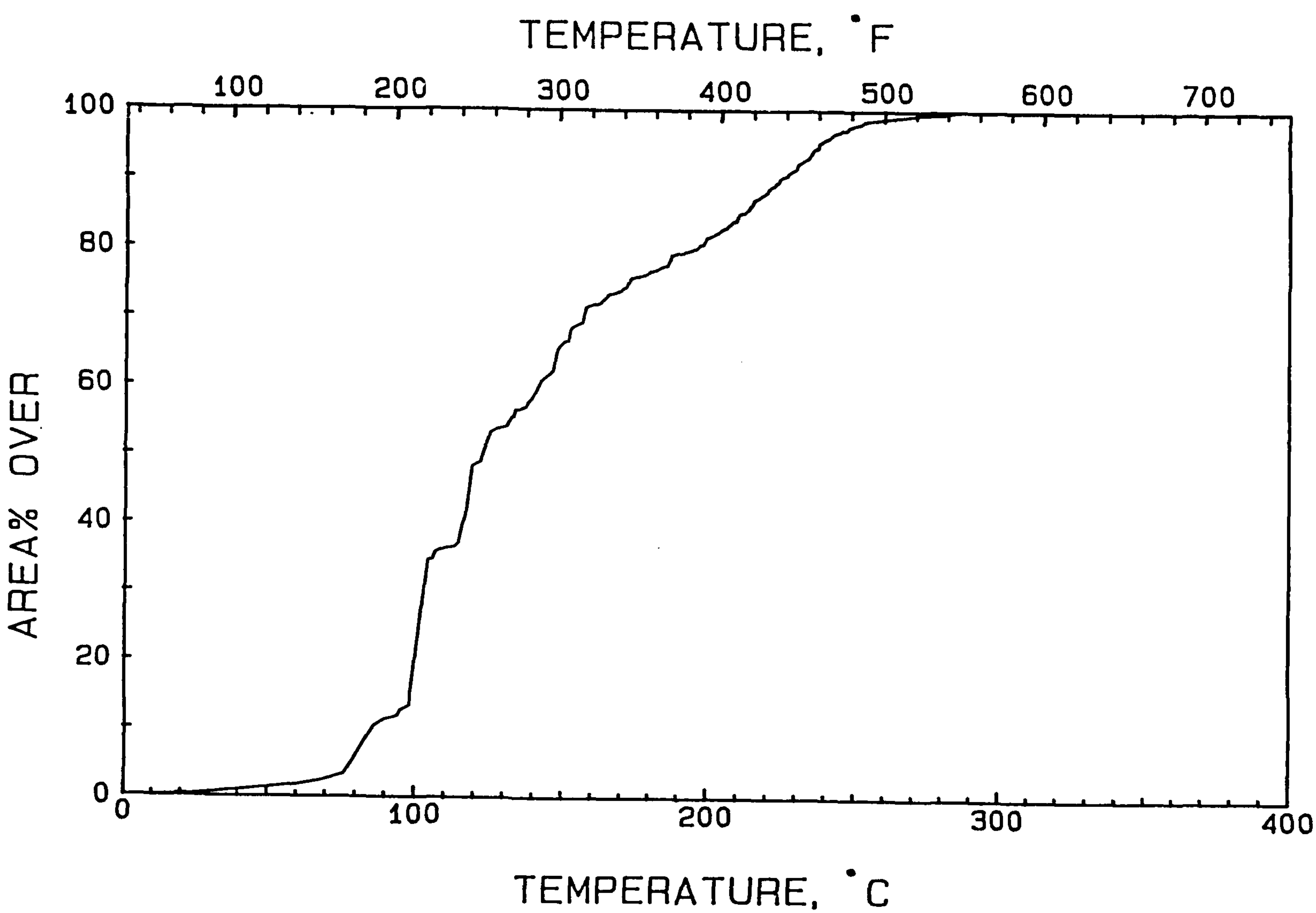

Figure 2. Simulated distillation data for coal-derived JP-4.

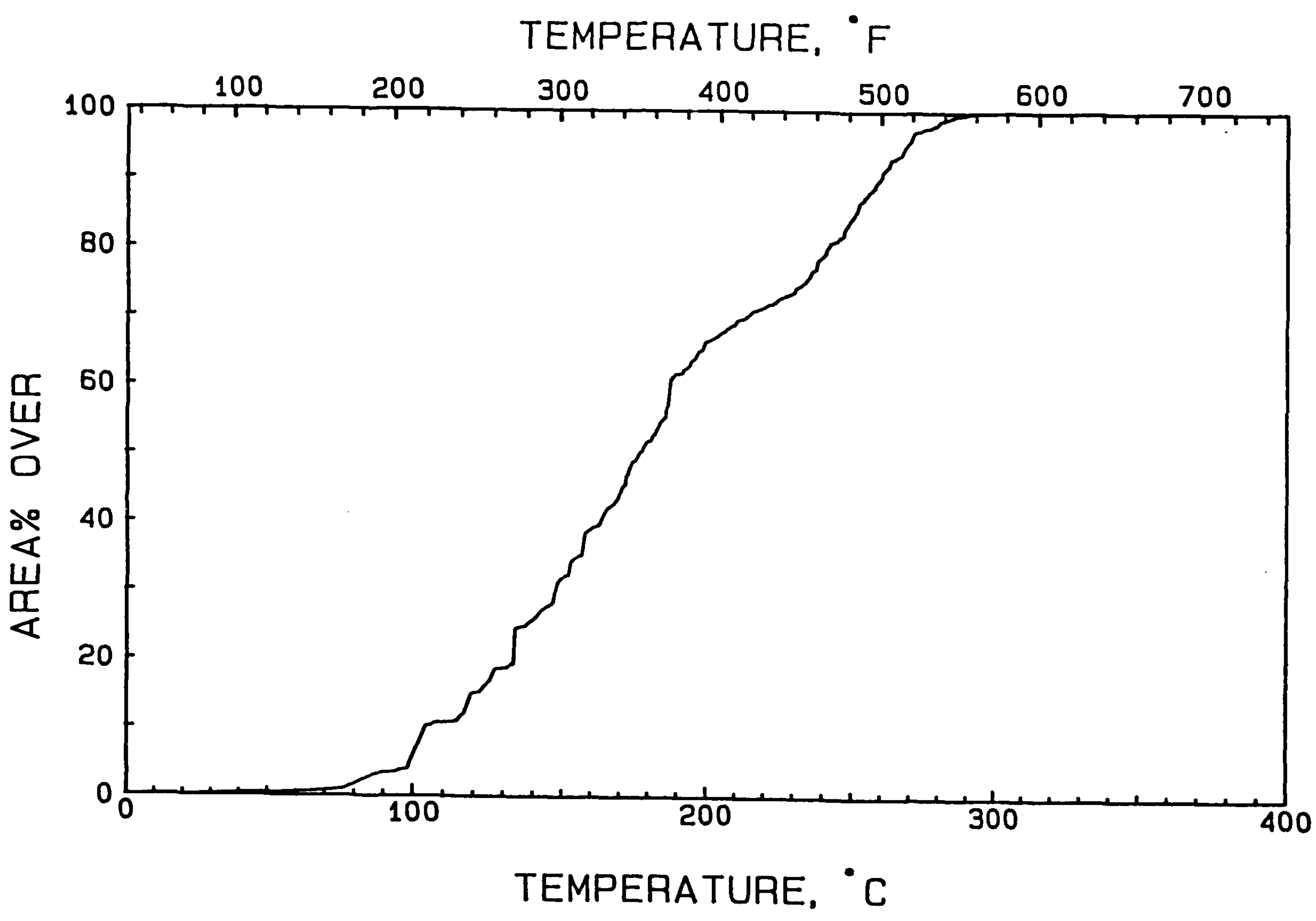

Figure 3. Simulated distillation data for coal-derived JP-8.

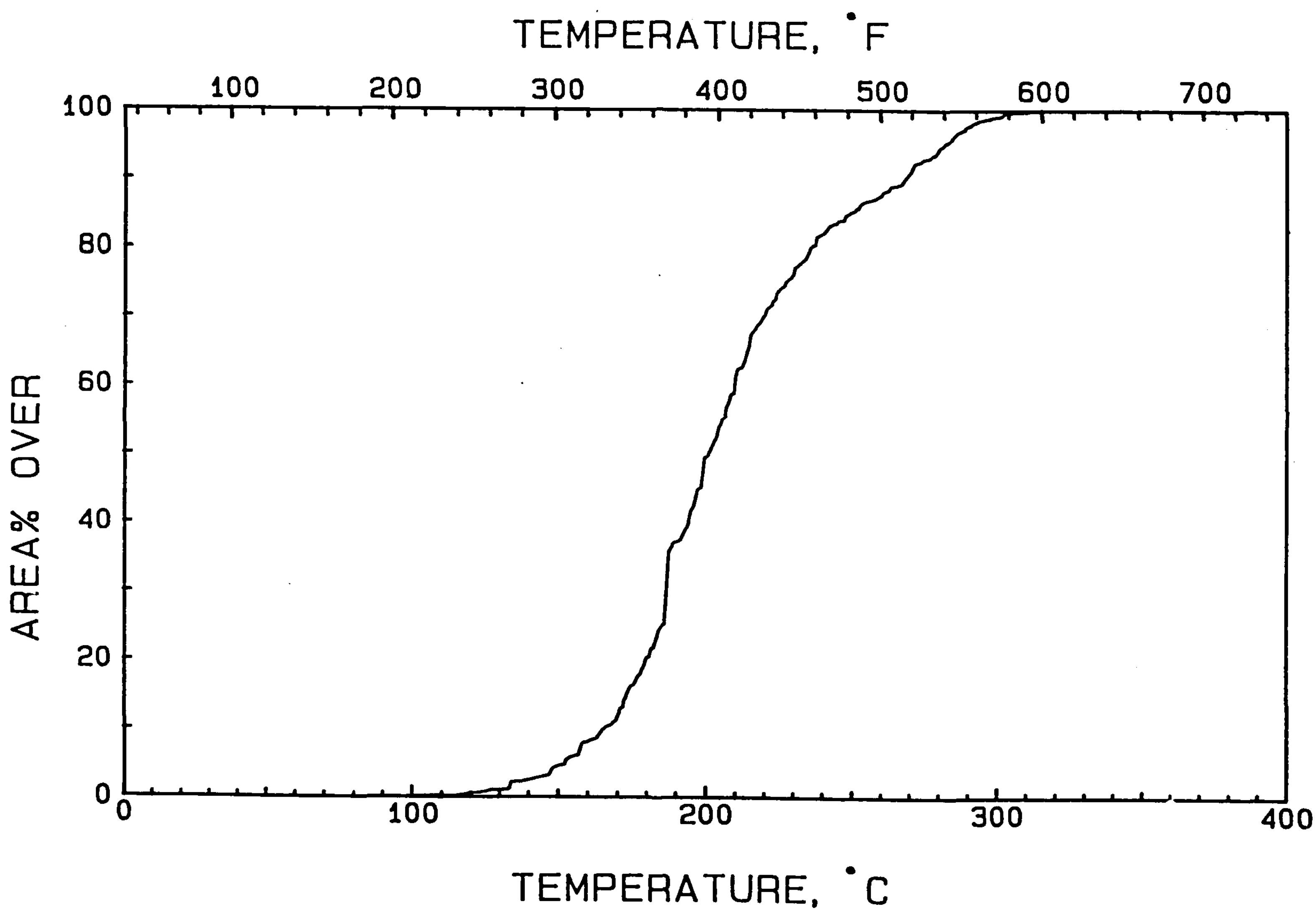

Figure 4. Simulated distillation data for coal-derived JP-8X.

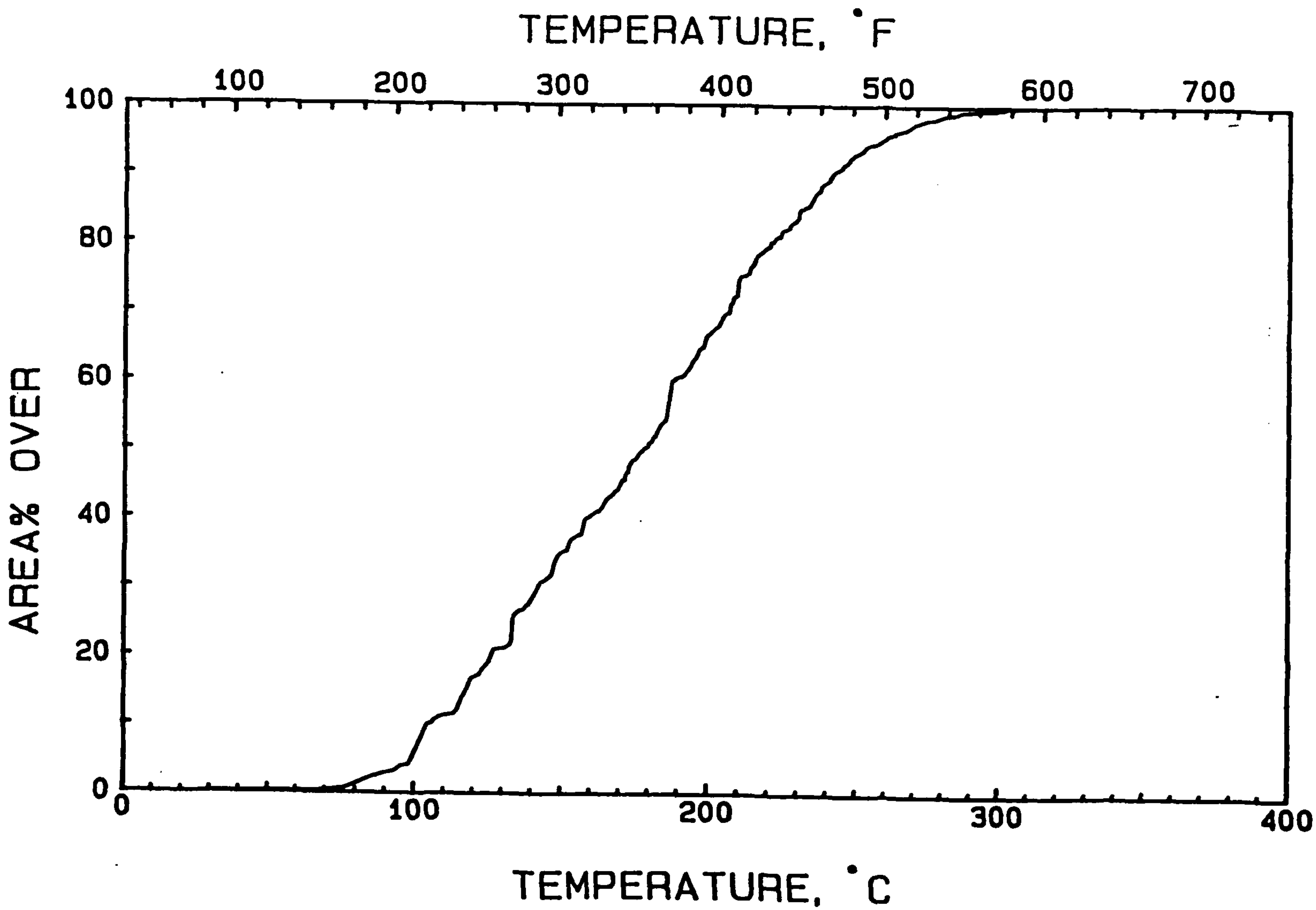

Figure 5. Simulated distillation data for bulk coal-derived JP-8.

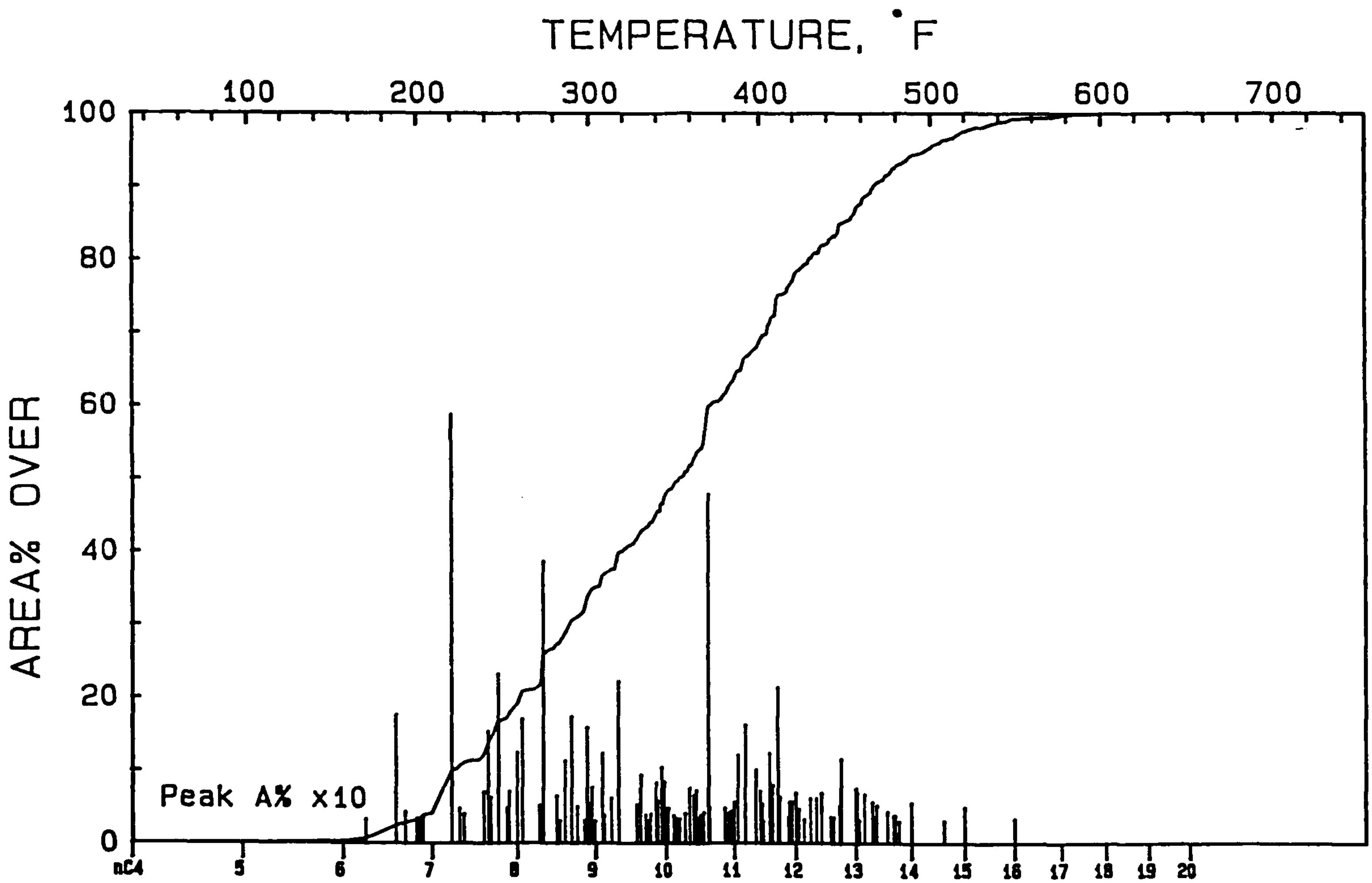

Figure 6. Relationship of simulated distillation data to GC component data.

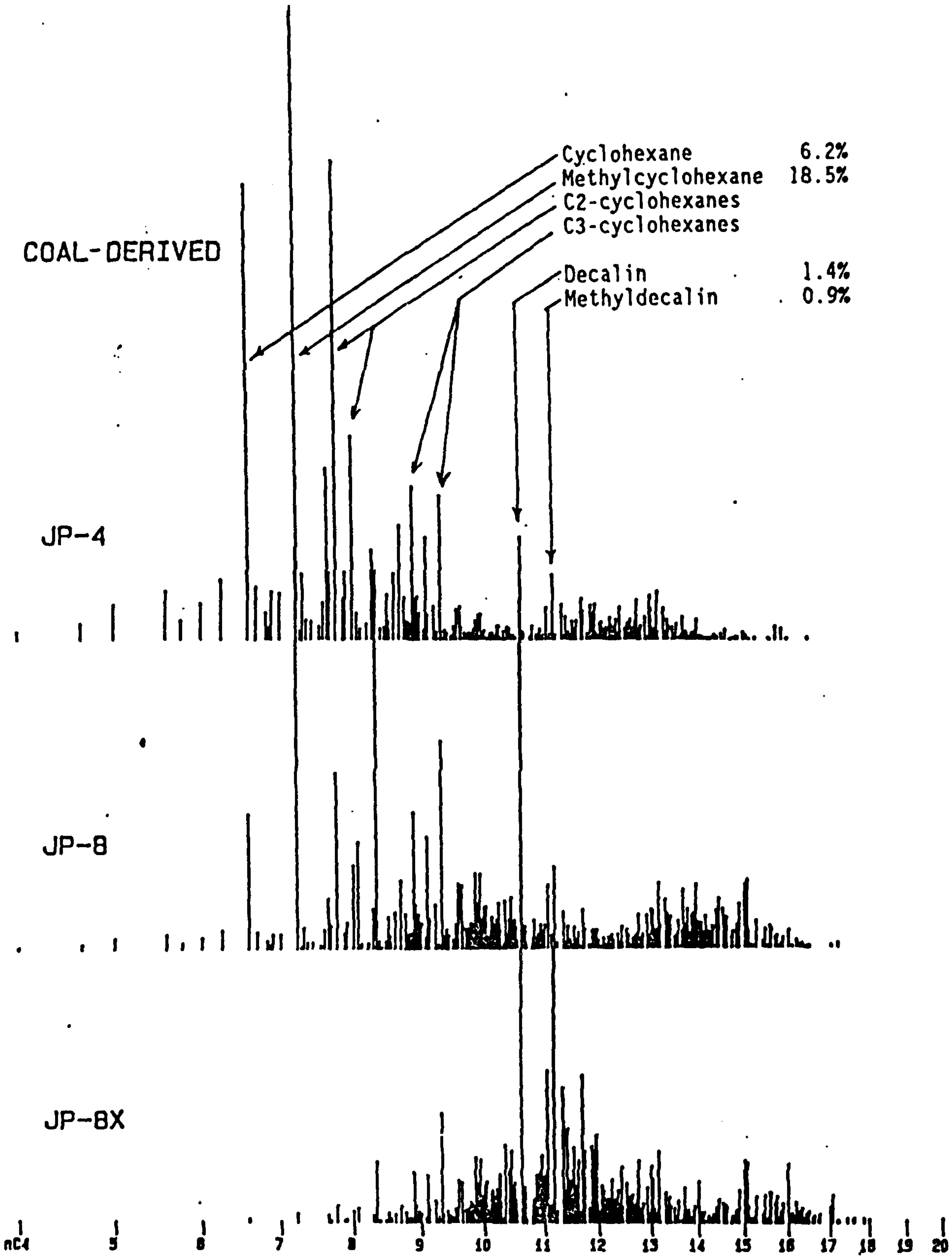

Figure 7. GC component data for coal-derived JP-4, JP-8, and JP-8X.

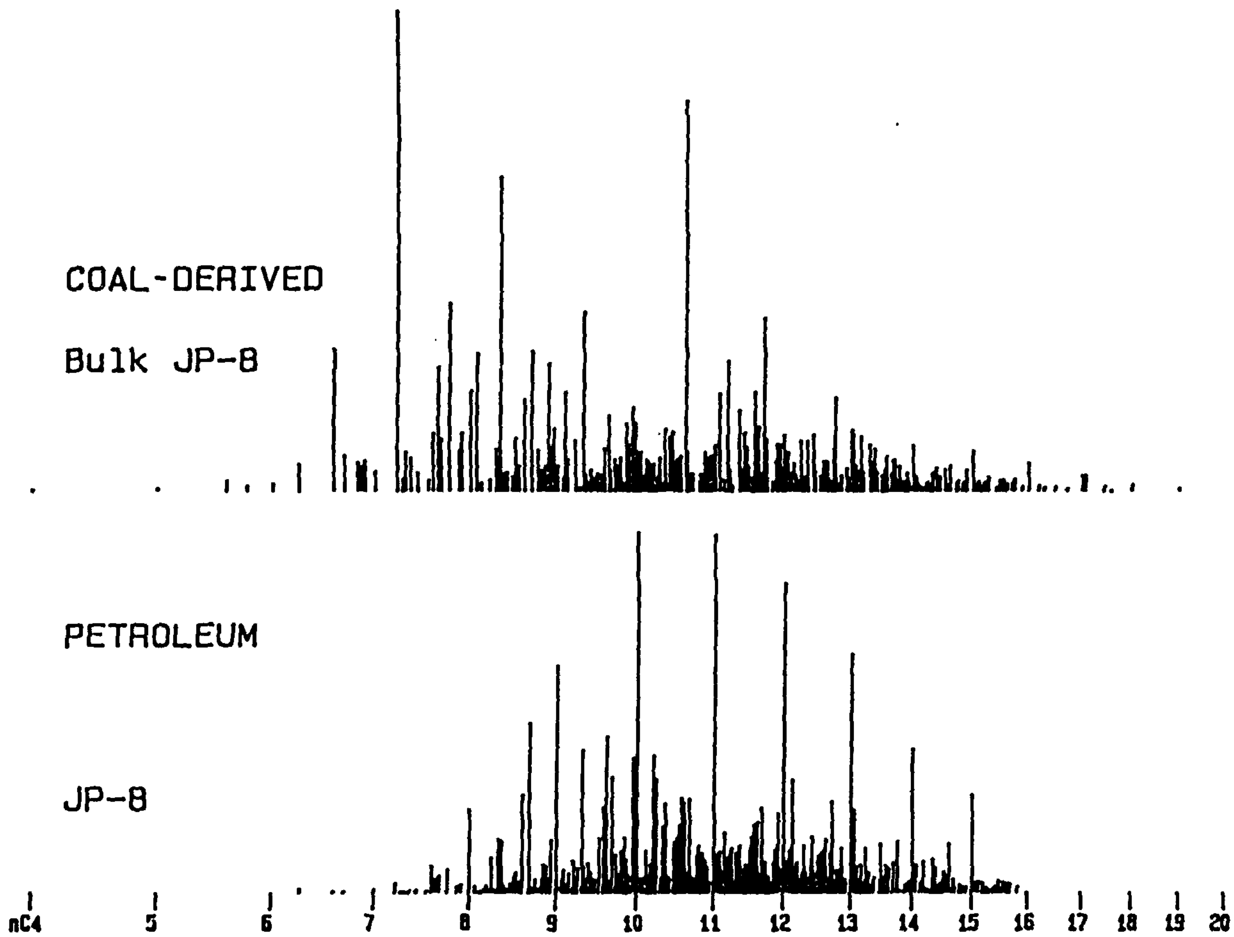

Figure 8. GC component data for coal-derived bulk JP-8 and petroleum-derived JP-8.

Coal-Derived JP-4, JP-8, and JP-8X

Initial samples were produced by Amoco in a bench scale unit in order to evaluate the overall upgrading process. In general, the tar oil stream was processed to yield hydrogenated product, which was fractionated by distillation and recombined to provide samples representative of JP-4, JP-8, and JP-8X jet fuels. Figures 2-4 depict the simulated distillation data, while Figure 7 and Table 3 present the GC component data. The component data indicates the similarities between the coal-derived jet fuels as well as the distillation cuts that were blended.

Figure 9 depicts ratioed component data of JP-4, JP-8, and JP-8X. The concentration of each GC component in JP-4 was divided by the analogous component in JP-8 and plotted as positive values. The JP-8X values were also divided by the analogous components in JP-8, multiplied by minus 1, and plotted. JP-8 was plotted at the bottom of the figure for comparative purposes. The ratio of light ends (C4-to-C7 region) qive a constant value followed by a smooth decrease, indicating that JP-4 and JP-8 were separated from the same total sample by distillation and some JP-4 was added back to the JP-8 fraction. The heavy ends (greater than C15) are only present in JP-8X. The smooth curve from C14 to C17 indicates that this heavy fraction was removed from the total sample by distillation and added to the JP-8X fraction. The ratio curves between C9 and C14 indicate a number of additional different distillation cuts were obtained from a total sample and blended by Amoco to meet fuel specifications and to optimize the yield structure. The coal-derived jet fuel contains different distillation cuts, resulting in a nonuniform component envelope (Figure 8, top), unlike the petroleum derived JP-8 which exhibits a smooth component envelope (Figure 8, bottom).

Coal-Derived Bulk JP-8

The coal-derived bulk JP-8 was produced in a pilot plant operation by Amoco Oil Company. The GC data for the coal-derived JP-8 is depicted in Figures 5 and 8 (top). In contrast to the petroleum-derived JP-8, there are only small amounts of straight-chained aliphatics present in the coal-derived JP-8. The base aliphatic functionalities in coal-derived jet fuel are cycloaliphatics (cyclohexane and decalin). The smaller background peaks are mainly alkylated cycloaliphatics. Unhydrotreated toluene and xylenes are present in higher concentrations than in petroleum JP-8.

ACKNOWLEDGEMENTS

The oxygenates analysis research at UNDEMRC was performed by Dr. Steven Hawthorne and David Miller. GC/FID/MS analyses were performed by Dr. Steven Hawthorne and David Miller and by Dr. Edwin Olson and John Diehl.

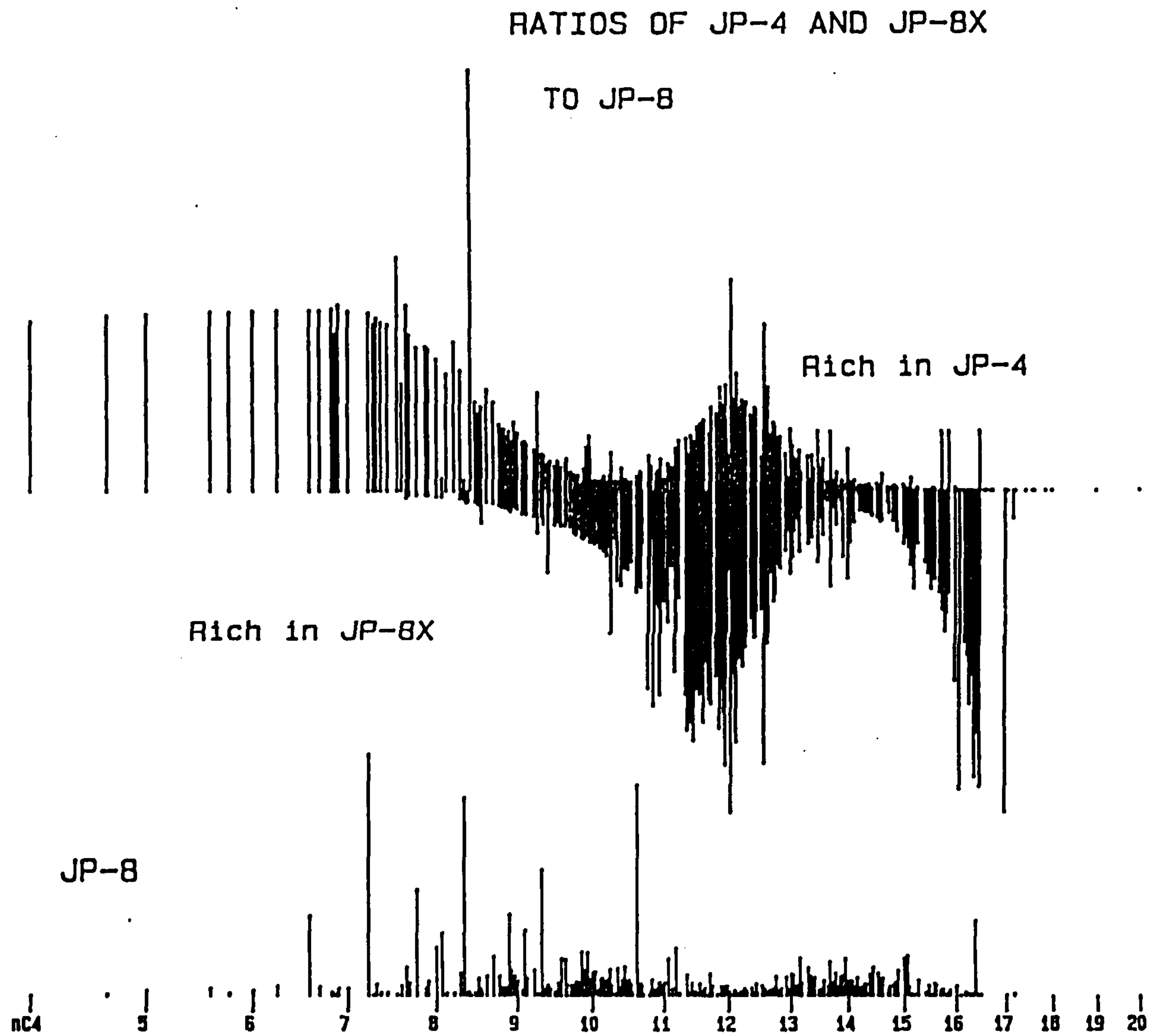

Figure 9. GC component difference data for coal-derived JP-4 and JP-8X to JP-8.

TABLE 3

GC/FID/MS DATA FOR COAL-DERIVED JET FUELS

#	Compound	RI	Rt	JP-4 wt%	JP-8 wt%	Bulk wt%	JP-8X wt%	Cumulative wt% JP-4	Bulk	JP-8X
1.	n-C4	400.0	0.001	0.11	0.04	0.02	.	0.11	0.02	.
2.		466.0	2.837	0.21	0.07	.	.	0.31	0.02	.
3.	n-C5	500.0	4.300	0.48	0.16	0.02	.	0.79	0.04	.
4.		560.0	4.840	0.67	0.22	0.13	.	1.46	0.17	.
5.		577.9	5.001	0.26	0.09	0.07	.	1.72	0.24	.
6.	n-C6	600.0	5.200	0.49	0.16	0.10	.	2.21	0.34	.
7.		625.5	5.506	0.83	0.27	0.34	.	3.04	0.67	.
8.	Cyclohexane	659.7	5.916	6.34	2.10	1.76	0.04	9.38	2.43	0.04
9.		670.1	6.042	0.73	0.24	0.44	.	10.11	2.87	0.04
10.	C7H14	682.8	6.193	0.36	0.12	0.35	.	10.47	3.22	0.04
11.	C7H14	686.4	6.237	0.18	0.07	0.30	.	10.66	3.52	0.04
12.	C7H14	689.8	6.277	0.67	0.21	0.37	.	11.32	3.90	0.04
13.	n-C7	700.0	6.400	0.64	0.21	0.24	.	11.97	4.13	0.04
14.	C1-Cyclohexane	723.0	7.952	18.79	6.32	5.85	0.13	30.75	9.98	0.17
15.		726.3	8.177	.	.	0.11	.	30.75	10.09	0.17
16.		728.5	8.321	0.12	0.04	0.15	.	30.87	10.24	0.17
17.	C2-Cyclopropane	731.8	8.549	0.91	0.31	0.48	.	31.78	10.73	0.17
18.	C3-Cyclopropane	736.9	8.890	0.27	0.10	0.41	.	32.06	11.14	0.17
19.	C3-Cyclopropane	744.3	9.391	0.24	0.09	0.23	.	32.30	11.37	0.17
20.	C3-Cyclopropane	755.3	10.134	0.17	0.04	0.13	.	32.47	11.50	0.17
21.	C8H18	760.5	10.483	0.51	0.28	0.71	.	32.98	12.21	0.17
22.	Toluene	765.7	10.837	2.37	0.76	1.54	0.11	35.35	13.74	0.28
23.		769.1	11.062	0.93	0.36	0.63	0.03	36.29	14.38	0.31
24.	C2-Cyclohexane	777.6	11.638	6.67	2.76	2.31	0.26	42.95	16.69	0.57
25.	C3-Cyclopropane	787.8	12.328	0.58	0.24	0.49	0.02	43.53	17.18	0.59
26.	C3-Cyclopropane	790.6	12.513	0.93	0.39	0.71	0.04	44.47	17.90	0.63
27.	n-C8	800.0	13.150	2.85	1.28	1.25	0.16	47.31	19.14	0.80
28.	C2-Cyclohexane	806.9	13.339	3.59	1.64	1.70	0.21	50.90	20.84	1.01
29.		812.1	13.483	0.14	.	0.10	.	51.04	20.94	1.01
30.		821.9	13.753	0.21	.	0.14	.	51.24	21.09	1.01
31.		829.3	13.957	1.23	0.61	0.52	0.11	52.47	21.60	1.12
32.	C9H18	831.6	14.018	.	.	0.39	0.06	52.47	22.00	1.18
33.	C2-Cyclohexane	834.3	14.093	9.53	5.17	3.84	0.96	62.00	25.84	2.14
34.		837.1	14.170	.	0.09	0.20	0.02	62.00	26.05	2.16
35.		839.2	14.228	.	0.11	0.22	0.03	62.00	26.27	2.19
36.		842.0	14.304	0.15	0.02	0.23	.	62.16	26.50	2.19
37.	C3-Cyclohexane	847.8	14.464	0.17	0.12	0.18	0.03	62.33	26.68	2.22
38.	C1-Octane	851.7	14.573	0.62	0.47	0.65	0.14	62.95	27.33	2.36
39.		854.2	14.641	0.25	0.20	.	.	63.20	27.33	2.36
40.		855.6	14.678	0.15	0.11	0.31	0.07	63.36	27.65	2.42
41.		858.3	14.754	0.91	0.54	0.12	0.03	64.27	27.77	2.45
42.	C2-Benzene	862.1	14.858	0.05	0.04	1.13	0.12	64.32	28.90	2.57
43.	m+p-Xylene	870.2	15.082	1.56	1.04	1.73	0.25	65.89	30.64	2.83
44.	C3-cyclohexane	877.4	15.279	0.58	0.53	0.51	0.16	66.46	31.14	2.98
45.	C3-Cyclohexane	880.9	15.376	0.24	0.23	0.26	0.08	66.71	31.40	3.06
46.		884.0	15.459	0.24	0.24	0.25	0.08	66.95	31.66	3.14
47.	C3-Cyclohexane	886.1	15.518	0.22	0.25	0.31	0.09	67.17	31.97	3.23
48.	C3-Cyclohexane	889.7	15.617	2.11	2.13	1.58	0.80	69.29	33.55	4.02
49.	C2-Benzene	891.8	15.674	0.57	0.68	0.54	0.27	69.86	34.09	4.30
50.	o-Xylene	896.0	15.789	0.60	0.53	0.77	0.17	70.46	34.86	4.47
51.	n-C9	900.0	15.900	0.38	0.39	0.31	0.14	70.83	35.17	4.62
52.	C3-Cyclohexane	906.5	16.063	0.14	0.18	0.17	0.08	70.97	35.34	4.70
53.	C3-Cyclohexane	909.5	16.138	1.40	1.73	1.23	0.75	72.38	36.57	5.45
54.	C9H18	912.2	16.204	0.35	0.45	0.39	0.21	72.72	36.96	5.66
55.	C9H18	922.2	16.455	0.46	0.67	0.62	0.34	73.18	37.58	6.00

TABLE 3 (cont.)

GC/FID/MS DATA FOR COAL-DERIVED JET FUELS

#	Compound	RI	Rt	JP-4 wt%	JP-8 wt%	Bulk wt%	JP-8X wt%	Cumulative wt% JP-4	Bulk	JP-8X
56.		927.0	16.576	0.12	0.07	0.05	0.06	73.30	37.63	6.06
57.	C3-Cyclohexane	932.3	16.708	1.98	3.26	2.21	1.74	75.28	39.84	7.80
58.		934.8	16.771	0.12	0.21	0.21	0.14	75.40	40.05	7.94
59.	C4-Cyclohexane	939.6	16.889	0.13	0.29	0.12	.	75.54	40.16	7.94
60.	C4-Cyclohexane	941.2	16.930	0.05	0.12	0.27	0.18	75.59	40.43	8.12
61.	C4-Cyclohexane	943.6	16.991	0.10	0.21	0.14	0.07	75.69	40.57	8.19
62.	C4-Cyclohexane	945.4	17.035	.	.	0.16	0.13	75.69	40.73	8.31
63.		949.5	17.137	0.06	0.15	0.22	0.10	75.75	40.95	8.41
64.	C3-Benzene	953.7	17.242	0.18	0.37	0.21	0.22	75.93	41.16	8.63
65.		958.5	17.363	0.42	1.00	0.53	0.67	76.35	41.69	9.30
66.	C3-Benzene	963.9	17.498	0.46	0.97	0.94	0.65	76.81	42.63	9.95
67.	C3-Benzene	965.6	17.541	0.17	0.32	0.27	0.19	76.98	42.90	10.14
68.	C3-Benzene	970.7	17.668	0.10	0.31	0.40	0.21	77.08	43.30	10.35
69.		975.4	17.784	0.09	0.32	0.32	0.25	77.17	43.62	10.61
70.		978.0	17.850	0.10	0.39	0.41	0.32	77.27	44.03	10.93
71.		981.3	17.933	.	0.38	0.11	0.29	77.27	44.14	11.22
72.	C4-Cyclohexane	985.6	18.039	0.16	1.16	0.84	1.04	77.43	44.98	12.26
73.	C4-Cyclohexane	988.5	18.114	0.27	0.71	0.58	0.64	77.70	45.56	12.90
74.		992.1	18.202	0.16	0.22	.	0.18	77.87	45.56	13.08
75.		993.3	18.234	0.35	1.15	1.04	0.99	78.21	46.60	14.07
76.	C3-Benzene	996.1	18.303	0.35	0.38	.	0.31	78.56	46.60	14.39
77.		997.2	18.329	0.12	0.47	0.85	0.46	78.68	47.45	14.85
78.	n-C10	1000.0	18.400	0.10	0.63	0.48	0.60	78.78	47.93	15.44
79.		1003.2	18.474	0.12	0.64	0.48	0.65	78.91	48.41	16.09
80.	C1-BCN	1007.8	18.579	0.04	0.22	0.19	0.21	78.94	48.60	16.30
81.	C1-BCN	1010.8	18.650	.	0.44	0.39	0.47	78.94	48.99	16.77
82.	C1-BCN	1013.1	18.700	0.09	0.47	0.36	0.51	79.03	49.35	17.28
83.	C1-BCN	1016.3	18.775	0.10	0.39	0.34	0.45	79.13	49.69	17.73
84.		1018.3	18.822	.	0.07	0.07	0.07	79.13	49.76	17.80
85.		1020.4	18.869	0.07	0.44	0.34	0.54	79.20	50.10	18.35
86.		1024.2	18.956	0.18	0.69	0.17	0.73	79.39	50.28	19.08
87.	C4-Benzene	1026.7	19.014	0.18	0.29	0.20	0.77	79.57	50.48	19.85
88.	C4-Benzene	1028.1	19.046	0.05	.	0.41	0.33	79.62	50.89	20.19
89.	C4-Benzene	1032.0	19.136	0.02	0.09	0.10	0.09	79.64	50.99	20.27
90.	C10H20	1034.8	19.201	0.12	0.73	0.76	1.24	79.76	51.75	21.51
91.		1037.9	19.273	0.04	0.24	0.08	.	79.80	51.84	21.51
92.	Indane	1040.7	19.336	0.17	0.44	0.67	0.79	79.97	52.50	22.30
93.	C4-Benzene	1044.6	19.427	0.15	0.77	0.73	1.14	80.13	53.23	23.44
94.		1046.4	19.466	0.08	0.40	0.34	0.57	80.21	53.57	24.02
95.		1050.5	19.562	0.07	0.43	0.40	0.63	80.27	53.97	24.65
96.		1053.4	19.629	0.04	0.33	0.20	0.35	80.32	54.17	25.00
97.		1055.3	19.671	0.02	0.12	0.43	0.16	80.34	54.60	25.17
98.	t-Decalin	1062.3	19.832	1.40	5.52	4.76	10.46	81.74	59.37	35.63
99.		1063.6	19.864	.	0.09	0.49	0.17	81.74	59.85	35.79
100.		1067.0	19.941	.	0.11	0.21	0.57	81.74	60.06	36.37
101.		1068.5	19.975	.	0.27	0.15	0.49	81.74	60.22	36.85
102.		1070.5	20.021	0.08	0.07	0.21	0.07	81.82	60.43	36.92
103.		1079.1	20.219	0.04	0.07	0.12	0.27	81.86	60.55	37.20
104.		1080.8	20.257	0.05	0.18	0.22	0.30	81.91	60.77	37.50
105.		1082.7	20.302	.	0.43	0.16	0.39	81.91	60.92	37.89
106.		1086.7	20.393	0.19	0.19	0.48	0.76	82.10	61.41	38.65
107.		1089.1	20.449	0.07	0.23	0.25	0.44	82.17	61.66	39.09
108.	C1-Indane	1091.6	20.507	0.08	0.24	0.42	0.51	82.26	62.08	39.60
109.		1092.9	20.536	.	0.36	0.26	0.80	82.26	62.35	40.40
110.		1095.4	20.595	0.14	0.27	0.44	1.07	82.40	62.79	41.46

TABLE 3 (cont.)

GC/FID/MS DATA FOR COAL-DERIVED JET FUELS

#	Compound	RI	Rt	JP-4 wt%	JP-8 wt%	Bulk wt%	JP-8X wt%	Cumulative wt% JP-4	Bulk	JP-8X
111.	n-C11	1100.0	20.700	0.08	0.36	0.56	0.75	82.48	63.35	42.22
112.		1106.3	20.830	0.44	0.98	1.21	2.42	82.92	64.56	44.63
113.	C1-Decalin	1112.2	20.949	0.08	0.21	0.14	0.29	83.00	64.70	44.92
114.	C1-Decalin	1118.1	21.072	0.89	1.25	1.62	4.33	83.90	66.32	49.25
115.	c1-Decalin	1120.8	21.126	0.03	.	0.27	0.05	83.93	66.59	49.31
116.		1123.6	21.183	0.07	0.09	0.07	0.18	84.00	66.66	49.49
117.	C1-Decalin	1134.9	21.415	0.48	0.55	1.00	2.16	84.48	67.66	51.64
118.	C1-Decalin	1138.3	21.485	0.07	0.12	0.26	0.52	84.55	67.92	52.17
119.	C1-Decalin	1142.3	21.568	0.31	0.34	0.72	1.50	84.86	68.64	53.67
120.	C1-Decalin	1145.6	21.636	0.09	0.14	0.54	0.41	84.95	69.18	54.08
121.	C1-Decalin	1147.6	21.676	0.12	0.14	0.31	0.69	85.07	69.49	54.77
122.	C1-Decalin	1152.1	21.767	.	0.09	0.18	0.33	85.07	69.67	55.10
123.	C1-Decalin	1154.3	21.812	0.09	0.32	0.11	1.20	85.16	69.78	56.30
124.		1157.3	21.874	0.25	0.15	1.24	0.60	85.42	71.02	56.90
125.	C5-Benzene	1159.6	21.921	0.17	0.12	0.19	0.44	85.59	71.20	57.34
126.	C1-Indane	1162.6	21.983	0.11	0.23	0.80	1.00	85.70	72.01	58.34
127.		1168.1	22.096	0.26	0.06	0.24	0.21	85.96	72.24	58.54
128.	Tetralin	1170.7	22.148	0.57	0.59	2.13	2.35	86.53	74.38	60.89
129.	C2-Decalin	1174.1	22.220	0.40	0.28	0.64	1.15	86.92	75.01	62.04
130.		1180.0	22.340	0.05	.	0.11	0.14	86.97	75.12	62.18
131.		1181.5	22.371	0.03	0.07	0.11	0.25	87.00	75.23	62.43
132.		1183.9	22.419	0.09	.	0.04	0.12	87.10	75.27	62.55
133.	C2-Decalin	1187.6	22.497	0.47	0.27	0.37	1.22	87.56	75.64	63.76
134.	C2-Decalin	1189.6	22.536	0.28	0.19	0.58	0.73	87.85	76.22	64.50
135.	C2-Decalin	1194.1	22.628	0.38	0.27	0.57	1.15	88.23	76.79	65.65
136.	C2-Decalin	1196.4	22.676	0.48	0.27	0.24	1.40	88.71	77.03	67.04
137.		1197.6	22.700	0.11	0.09	0.28	0.33	88.82	77.31	67.38
138.	n-C12	1200.0	22.750	0.08	0.13	0.69	0.20	88.90	78.00	67.58
139.	C2-Decalin	1205.3	22.855	0.21	0.06	0.47	0.59	89.11	78.47	68.17
140.	C2-Decalin	1208.6	22.921	0.23	0.15	0.22	0.53	89.34	78.69	68.70
141.	C2-Undecane	1213.7	23.025	0.16	0.08	0.34	0.40	89.51	79.03	69.10
142.	C2-Decalin	1215.9	23.068	0.13	0.09	0.20	0.30	89.64	79.23	69.40
143.	C2-Decalin	1218.9	23.129	0.18	0.13	0.11	0.40	89.82	79.33	69.79
144.		1222.0	23.190	0.17	0.11	0.14	0.37	89.99	79.47	70.16
145.	C1-Tetralin	1224.7	23.244	0.31	0.21	0.62	0.69	90.30	80.09	70.85
146.	C2-Decalin	1228.7	23.324	0.20	0.13	0.18	0.40	90.50	80.27	71.25
147.	C3-Decalin	1234.6	23.441	0.18	0.07	0.62	0.38	90.68	80.89	71.63
148.		1236.0	23.470	0.28	0.22	.	0.52	90.96	80.89	72.16
149.		1240.5	23.561	0.13	0.10	.	0.27	91.09	80.89	72.43
150.		1243.1	23.611	0.44	0.32	0.69	0.89	91.53	81.58	73.32
151.		1245.3	23.656	0.14	0.11	0.24	0.24	91.67	81.82	73.56
152.	C1-Tetralin	1252.5	23.800	0.16	0.28	0.19	0.62	91.83	82.01	74.18
153.		1256.9	23.888	0.19	0.20	0.28	0.22	92.03	82.29	74.40
154.	C6-Benzene	1258.3	23.916	0.23	0.08	0.37	0.42	92.25	82.66	74.83
155.	C3-Decalin	1263.8	24.025	0.27	0.15	0.37	0.45	92.52	83.03	75.28
156.		1268.8	24.125	0.18	0.19	0.14	0.30	92.70	83.16	75.58
157.	C1-Dodecane	1272.9	24.207	0.26	0.23	0.52	0.47	92.96	83.68	76.05
158.	C1-Tetralin	1275.4	24.257	0.54	0.51	1.16	0.99	93.50	84.84	77.04
159.	C3-Decalin	1279.5	24.341	0.15	0.17	0.10	0.24	93.65	84.93	77.28
160.	C3-Decalin	1283.8	24.427	0.19	0.22	0.19	0.31	93.85	85.12	77.59
161.		1287.2	24.493	0.07	.	0.14	0.10	93.92	85.26	77.69
162.	C2-Tetralin	1291.7	24.584	0.31	0.50	0.28	0.57	94.23	85.54	78.26
163.		1294.3	24.637	0.05	0.22	0.17	0.16	94.28	85.70	78.42
164.	n-C13	1300.0	24.750	0.60	0.59	0.75	0.91	94.89	86.46	79.33
165.		1303.2	24.811	0.42	0.56	0.70	0.71	95.31	87.15	80.04

TABLE 3 (cont.)

GC/FID/MS DATA FOR COAL-DERIVED JET FUELS

#	Compound	RI	Rt	JP-4 wt%	JP-8 wt%	Bulk wt%	JP-8X wt%	Cumulative wt% JP-4	Bulk	JP-8X
166.	C2-Decalin	1306.2	24.869	0.16	0.29	0.32	0.22	95.47	87.48	80.26
167.	C2-Decalin	1312.1	24.980	0.16	0.30	0.26	0.17	95.63	87.74	80.43
168.		1315.1	25.037	0.66	1.00	0.67	1.13	96.29	88.41	81.56
169.		1317.8	25.089	0.11	0.36	0.24	0.12	96.40	88.65	81.68
170.	C12H22	1329.3	25.306	0.43	0.74	0.57	0.48	96.83	89.22	82.17
171.	C2-Tetralin	1330.7	25.333	.	0.22	0.36	0.21	96.83	89.58	82.38
172.		1333.2	25.380	0.11	0.53	0.20	0.14	96.94	89.78	82.53
173.	C2-Tetralin	1337.2	25.457	0.27	0.46	0.52	0.41	97.21	90.30	82.94
174.	C2-Tetralin	1340.3	25.515	0.18	0.48	0.25	0.28	97.39	90.55	83.21
175.	C2-Tetralin	1347.8	25.658	0.17	0.18	0.21	0.23	97.57	90.76	83.45
176.		1353.5	25.766	0.08	0.22	0.29	0.11	97.65	91.04	83.56
177.	TCTD	1356.5	25.824	0.21	0.40	0.43	0.34	97.86	91.47	83.89
178.		1363.5	25.956	0.05	0.28	0.21	0.06	97.91	91.68	83.95
179.	C2-Tetralin	1367.5	26.032	0.08	0.89	0.38	0.14	97.99	92.06	84.09
180.	C2-Tetralin	1370.1	26.081	0.30	0.31	0.38	0.56	98.29	92.45	84.65
181.	C2-Tetralin	1374.2	26.159	0.06	0.37	0.12	0.12	98.35	92.57	84.77
182.	C1-Tridecane	1377.2	26.218	0.11	0.59	0.31	0.20	98.46	92.88	84.97
183.		1380.1	26.273	0.08	0.25	0.11	0.16	98.54	92.98	85.13
184.	C2-Tetralin	1384.8	26.361	0.05	0.50	0.13	0.10	98.59	93.11	85.23
185.	C4-Decalin	1389.5	26.451	0.09	0.54	0.23	0.20	98.68	93.34	85.43
186.		1391.4	26.487	.	0.21	0.12	0.26	98.68	93.46	85.69
187.		1394.6	26.547	0.11	0.97	0.05	.	98.79	93.51	85.69
188.	n-C14	1400.0	26.650	0.28	0.40	0.56	0.66	99.06	94.07	86.35
189.		1402.3	26.687	.	.	.	.	99.06	94.07	86.35
190.		1404.1	26.717	.	0.18	0.18	0.18	99.06	94.25	86.53
191.		1406.2	26.752	0.06	0.39	0.09	0.07	99.12	94.34	86.60
192.		1411.1	26.834	0.05	0.26	0.05	0.16	99.17	94.39	86.76
193.		1415.0	26.898	0.03	0.48	0.04	0.03	99.20	94.43	86.79
194.	C3-Tetralin	1420.8	26.993	0.03	0.26	0.10	0.10	99.23	94.53	86.89
195.	C3-Tetralin	1423.0	27.030	0.02	0.22	0.11	0.08	99.25	94.64	86.97
196.	C3-Tetralin	1426.6	27.089	0.01	0.20	0.08	0.08	99.26	94.72	87.05
197.		1428.0	27.113	0.02	0.33	.	0.07	99.28	94.72	87.12
198.	C3-Tetralin	1431.2	27.165	0.03	0.32	0.23	0.13	99.31	94.95	87.25
199.	C3-Tetralin	1438.1	27.278	0.06	0.58	0.29	0.24	99.37	95.24	87.49
200.	C3-Tetralin	1441.7	27.338	.	0.34	0.15	0.15	99.37	95.39	87.64
201.	C3-Tetralin	1443.8	27.373	0.04	0.75	0.18	0.36	99.41	95.57	88.00
202.	C3-Tetralin	1452.7	27.519	0.08	0.60	0.27	0.31	99.49	95.84	88.31
203.	C3-Tetralin	1456.1	27.576	0.02	0.20	0.07	0.07	99.51	95.92	88.38
204.	C3-Tetralin	1458.5	27.615	.	0.48	0.05	0.28	99.51	95.96	88.66
205.	C3-Tetralin	1461.1	27.658	0.13	0.46	0.31	0.26	99.64	96.28	88.92
206.		1470.6	27.816	0.01	0.18	0.10	0.04	99.65	96.38	88.97
207.	C3-Tetralin	1473.9	27.870	0.02	0.26	0.13	0.11	99.67	96.51	89.08
208.		1481.5	27.995	0.02	0.31	0.09	0.18	99.69	96.60	89.26
209.		1483.9	28.035	0.03	0.36	0.12	0.21	99.72	96.72	89.47
210.	TCTD	1488.6	28.111	0.04	0.67	0.26	0.52	99.77	96.98	89.99
211.	n-C15	1500.0	28.300	0.10	0.98	0.50	0.99	99.86	97.48	90.98
212.		1506.4	28.408	0.06	1.05	0.08	0.96	99.92	97.56	91.94
213.	TDHP	1509.0	28.452	.	0.24	0.12	0.26	99.92	97.68	92.20
214.	TDHP	1513.6	28.532	0.01	0.06	0.07	0.08	99.94	97.76	92.28
215.	TDHP	1519.6	28.634	.	0.05	0.12	0.08	99.94	97.88	92.36
216.	TCTD	1526.9	28.758	0.02	0.40	0.18	0.41	99.96	98.05	92.77
217.		1541.4	29.003	.	0.10	0.05	0.14	99.96	98.11	92.90
218.		1546.8	29.095	.	0.28	0.14	0.44	99.96	98.25	93.34
219.		1553.2	29.204	.	0.05	0.15	0.10	99.96	98.40	93.44
220.	C1-TCTD	1558.0	29.286	0.02	0.33	0.10	0.49	99.97	98.50	93.92

TABLE 3 (cont.)

GC/FID/MS DATA FOR COAL-DERIVED JET FUELS

#	Compound	RI	Rt	JP-4 wt%	JP-8 wt%	Bulk wt%	JP-8X wt%	Cumulative wt% JP-4	Bulk	JP-8X
221.		1561.0	29.337	0.01	0.29	0.08	0.48	99.99	98.58	94.40
222.		1569.1	29.475	.	0.17	0.10	0.32	99.99	98.68	94.72
223.		1572.0	29.525	.	0.04	.	.	99.99	98.68	94.72
224.	C4-Tetralin	1574.4	29.565	.	0.19	0.15	0.42	99.99	98.83	95.13
225.		1579.4	29.649	.	0.04	.	0.10	99.99	98.83	95.24
226.	C4-Tetralin	1582.7	29.706	.	0.08	.	0.18	99.99	98.83	95.42
227.		1587.8	29.792	.	0.15	0.06	0.30	99.99	98.88	95.72
228.		1592.3	29.869	.	.	.	.	99.99	98.88	95.72
229.	n-C16	1600.0	30.000	0.02	0.26	0.34	0.93	100.00	99.23	96.65
230.		1605.8	30.098	.	.	.	0.13	100.00	99.23	96.78
231.		1608.9	30.151	.	0.03	.	0.15	100.00	99.23	96.92
232.		1612.4	30.211	.	.	.	0.11	100.00	99.23	97.03
233.		1616.7	30.284	.	.	.	0.10	100.00	99.23	97.14
234.		1618.4	30.313	.	0.11	0.07	0.31	100.00	99.29	97.45
235.		1625.0	30.425	.	0.06	.	0.18	100.00	99.29	97.63
236.		1629.2	30.496	.	0.03	0.04	0.13	100.00	99.34	97.77
237.		1632.8	30.557	.	0.06	.	0.22	100.00	99.34	97.99
238.		1638.8	30.660	.	0.02	.	0.11	100.00	99.34	98.10
239.		1643.4	30.737	.	0.03	.	0.15	100.00	99.34	98.25
240.		1649.4	30.840	.	0.03	0.05	0.17	100.00	99.39	98.42
241.		1657.9	30.984	.	.	.	0.09	100.00	99.39	98.51
242.		1662.7	31.065	.	.	.	0.11	100.00	99.39	98.62
243.		1672.0	31.224	.	.	0.03	0.07	100.00	99.41	98.69
244.		1675.3	31.280	.	.	.	0.12	100.00	99.41	98.81
245.		1678.2	31.329	.	.	.	.	100.00	99.41	98.81
246.		1682.9	31.409	.	.	.	.	100.00	99.41	98.81
247.		1686.4	31.469	.	.	.	.	100.00	99.41	98.81
248.	n-C17	1700.0	31.700	.	0.03	0.19	0.25	100.00	99.60	99.06
249.		1706.6	31.812	.	.	0.20	0.44	100.00	99.81	99.50
250.		1715.7	31.967	.	.	.	.	100.00	99.81	99.50
251.		1718.0	32.006	.	0.07	.	0.04	100.00	99.81	99.53
252.		1722.0	32.075	.	.	.	0.03	100.00	99.81	99.57
253.		1744.3	32.453	.	.	0.06	0.06	100.00	99.87	99.63
254.		1758.6	32.695	.	.	0.02	0.05	100.00	99.89	99.68
255.		1762.7	32.765	.	.	.	0.07	100.00	99.89	99.75
256.		1786.6	33.172	.	.	.	0.06	100.00	99.89	99.81
257.	n-C18	1800.0	33.400	.	.	0.08	0.05	100.00	99.97	99.86
258.	n-C19	1900.0	35.100	.	.	0.03	0.03	100.00	100.00	99.89
259.	n-C20	2000.0	36.800	.	.	.	0.06	100.00	100.00	99.95
260.	n-C21	2100.0	38.500	.	0.05	.	0.05	100.00	100.00	100.00

RI = GC column retention index; Rt = GC retention time (min); GC area% assumed equal to wt%.

LIST OF ABBREVIATIONS

amu	atomic mass units
area%	area percent
BCN	trans-bicyclo(4.0.3)nonane
BTX	mixture of benzene, toluene, and xylenes
C6, C7, etc.	normal alkanes containing 6, 7, etc. carbon atoms
°C	degrees Celsius
°F	degrees Fahrenheit
FID	flame ionization detector
GC	gas chromatography
GC/FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography with a mass spectrometer
JP-4	jet propulsion fuel, specification 4
JP-8	jet propulsion fuel, specification 8
JP-8X	jet propulsion fuel, specification 8X
m	meter
ml or mL	milliliters
N	normal
ND	not detected
nL	nanoliters
nm	nanometer
NMR	nuclear magnetic resonance
%	percent
ppm	parts per million
Rf	Response factor
Ri	retention index
SIM	selective ion monitoring
TCTD	tricyclotetradecane
TDHP	tetradecahydrophenanthrene
uL	microliters
UNDEERC	University of North Dakota Energy and Environmental Research Center formerly UNDEMRC
UNDEMRC	University of North Dakota Energy and Mineral Research Center
vol%	volume percent
WRI	Western Research Institute
wt%	weight percent

APPENDIX A

Energy & Mineral Research Center

Fuels & Process Chemistry Research Institute
ND Mining & Mineral Resources Research Institute
Combustion & Environmental Systems Research Institute

Box 8213, University Station / Grand Forks, North Dakota 58202 / Phone: (701) 777-5000 / Fax: 777-5181

March 14, 1988

Mr. Everette Harris
Senior Process Engineer
Hydrocarbon Research, Inc.
P.O. Box 6047
New York & Puritan Avenues
Lawrenceville, New Jersey 08648

SUBJECT: Contract No. DE-AC22-84PC72571, HRI Samples

Dear Sir:

The analyses of the eight HRI samples of the Great Plains phenolics stream which were treated in the Dynaphen Process have been completed. The data for the samples that were received for analysis are attached as Tables 1 through 12. In order to properly calculate this data, a computer program was completed to enable the data reduction (i.e., to match similar compounds and to calculate wt% values using response factors). Since this program has been developed, the data reduction of future GC data will be easy.

Table 1 presents the data for the samples as they were received and analyzed at UNDEMRC in comparison to values obtained by HRI. In general, the data is in fair agreement. Of the five samples containing isopropanol, two are essentially the same while the others are plus or minus about 10%. The water values for samples 237-50 and 237-51 indicate a larger difference while the other six values are similar.

The total peaks present in the GC output are indicated in Table 1 as well as the number of peaks that are over 0.1 A% (A% = area%). The sum of the total peaks is 100 A%, while the sums of the peaks of over 0.1 A% greater than 97%, indicating that the small peaks do not contain much material. The difference in the number of peaks is related to the amount of the sample that was injected to the GC. In addition, some samples contain less of certain compounds. Compounds in low concentrations may not be observed. In Tables 2 through 12, a period indicates a peak was not observed.

GC equipment used and conditions were as follows.

GC:	HP Model 5890
MS:	HP Model 5985B

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University Of North Dakota

Column:	DB-5, fused silica, 5% phenyl-methylsilicon bonded phase, film thickness 0.25 micron, 0.25 mm I.D., 60 m long.
Injected:	0.02 microliters, neat
Conditions:	0°C for 2 min followed by 6°C / min to 320°C

Table 2 presents a summary of the data for the HRI5511, 237-61RC, and L-731 samples. The KF-water values were determined by Karl Fischer analyses and calculated into the GC data to obtain as-received analyses values. The latter two samples contained larger quantities of heavier material that does not show up on this printout. If requested, a more complete printout can be obtained. Calculations were performed using 100 * Kwt% / Uwt% = Amount left where Kwt% is the value for a compound in the HRI5511 sample and Uwt% is the value in the L-731 sample. These calculations indicate that the L-731 sample represents 63 wt% of the HRI5511 sample in which the heavier material has been concentrated.

Table 3 presents a summary of the data, on an isopropanol-free basis, for the samples that initially contained isopropanol. Table 4 presents data calculated to be equal to the total liquid yield in the HRI letter dated Jan 6, 1988. Benzene and phenol values in the table are quite different from those mentioned in the letter. A cursory evaluation indicates that different response factors may have been used in the calculations. The raw GC data for the samples (including response factors) is given in Tables 5 through 12 for comparison with your data.

If you have any questions or suggestions on how to best present these data, please let me know.

Sincerely,

Curtis L. Knudson

Curtis L. Knudson, Ph.D.
Senior Research Associate
phone (701) 777-5185

HRI/clk

c: G.L. Stiegel (PETC)
W.E. Harrison (AFWAL-DOD)
D.P. Daley (B&RSC)
M.W. Furlong (Amoco)
A.K. Kuhn (ANG)
E.B. Smith (WRI)
W.G. Willson (UNDEMRC)

TABLE 1

ANALYSES OF AS-RECEIVED DYNAPHEN SAMPLES[a]

Sample ID	Isopropanol, wt% HRI	Isopropanol, wt% EMRC	Water, wt% HRI	Water, wt% EMRC	# GC Peaks Total	# GC Peaks over 0.1 A%	Total[b] A%
HRI5511	0.00	0.00	4.92	5.45	124	59	96.9
237-45	35.55	31.84	8.73[c]	9.23[d]	46	23	98.7
237-46	41.51	42.22	12.52[c]	11.98[e]	44	17	98.6
237-50	33.57	37.29	15.48	13.87	104	31	97.6
237-51	47.23	44.91	10.29	13.37	32	27	99.6
237-61	50.03	49.72	10.34	10.51	80	21	98.5
237-61RC	0.00	0.00	NA[f]	0.17[g]	147	53	95.7
L-731	0.00	0.00	0.1[g]	0.09[g]	168	63	95.7

[a]All values are for the as-received (AR) samples. Samples with a positive isopropanol value were received with the isopropanol present. HRI values were obtained from letters sent with the samples or verbally.
[b]This is the total A% (A% = Area%) in the peaks which were over 0.1 A%.
[c]Calculated from data which was not obtained by analysis.
[d]The average of duplicate analyses (9.03 and 9.43) obtained on different days.
[e]The average of duplicate analyses (12.25 and 11.72) obtained on different days.
[f]Not analyzed.
[g]Values for Karl Fischer water analyses of around 0.1 wt% are within error of zero.

TABLE 2

GC/MS SUMMARY DATA, ISOPROPANOL-FREE IN WT%

#	Compound / Sample	HRI5511	237-61RC	L-731
1.	KF-Water	5.45	0.17	0.09
2.	Isopropanol	.	.	.
3.	Benzene	.	0.03	.
4.	Toluene	0.16	39.22	.
5.	C2-Benzene	0.08	1.69	.
6.	mp-Xylene	0.12	2.22	.
7.	o-Xylene	0.07	0.14	0.01
8.	Aniline	0.13	0.21	0.28
9.	Phenol	44.55	2.14	25.38
10.	C3-Benzene	0.04	.	0.05
11.	Indane	0.02	.	.
12.	Indene	0.05	.	.
13.	o-Cresol	7.30	0.41	7.87
14.	mp-Cresol	18.22	14.25	29.11
15.	Guaiacol	1.65	.	2.63
16.	C2-Phenol	0.30	.	0.44
17.	C2-Phenols	1.34	0.13	0.01
18.	C2-Phenol	0.98	.	2.42
19.	C2-Phenol	1.38	1.10	2.24
20.	C2-Phenol	2.36	.	3.82
21.	Naphthalene	0.26	7.22	0.38
22.	C1-Guaiacol	0.08	0.22	0.13
23.	Catechol	1.59	0.10	2.66
24.	C3-Phenol	0.16	0.01	0.20
25.	C3-Phenol	0.18	.	0.29
26.	Quinoline	0.03	1.49	0.07
27.	C1-Catechol	1.76	0.07	2.76
28.	C3-Phenol	0.17	0.19	0.19
29.	C3-Phenol	0.16	.	0.18
30.	2-Methylnaphthalene	0.22	1.43	0.20
31.	1-Methylnaphthalene	0.24	0.45	0.39
32.	C2-Catechol	0.87	0.03	1.35
33.	Biphenyl	0.05	2.21	0.07
34.	C2-Catechol	0.91	0.02	1.62
35.	Acenaphthene	0.03	0.94	0.05
36.	Dibenzofuran	0.06	1.25	0.10
37.	Naphthol	0.41	1.46	0.61
38.	Phenanthrene	0.08	2.75	0.11
39.	Fluoranthene	0.02	0.69	0.02
40.	Pyrene	0.02	0.53	0.03
	Total wt%	91.51	82.77	85.73
	# of GC Peaks	38	30	33

KF-Water = Karl Fischer water.

TABLE 3

GC/MS SUMMARY DATA, ISOPROPANOL-FREE IN WT%

#	Compound / Sample	237-45	237-46	237-50	237-51	237-61
1.	KF-Water	13.61	20.89	22.36	24.47	21.15
2.	Isopropanol	.	.	.	.	.
3.	Benzene	11.43	31.39	6.49	14.76	27.26
4.	Toluene	4.98	3.97	6.74	7.31	10.06
5.	C2-Benzene	0.36	0.09	0.78	0.42	0.28
6.	mp-Xylene	0.52	0.09	1.02	0.53	0.32
7.	o-Xylene	0.08	.	0.22	0.08	0.05
8.	Aniline	.	0.15	0.27	.	0.27
9.	Phenol	54.61	35.29	35.73	37.87	28.21
10.	C3-Benzene	.	.	0.01	.	.
11.	Indane	0.17	0.08	0.29	0.22	0.14
12.	Indene	0.12	0.07	0.16	0.14	0.09
13.	o-Cresol	1.31	0.13	3.24	0.86	0.34
14.	mp-Cresol	7.91	1.15	15.27	6.76	3.90
15.	Guaiacol	.	.	.	.	.
16.	C2-Phenol	.	.	0.10	.	.
17.	C2-Phenols	0.07	.	0.31	.	0.01
18.	C2-Phenol	0.11	.	0.38	.	0.04
19.	C2-Phenol	.	.	0.30	.	0.16
20.	C2-Phenol	0.50	.	1.34	0.29	.
21.	Naphthalene	1.54	2.95	1.26	2.64	3.82
22.	C1-Guaiacol	.	0.05	0.03	.	0.08
23.	Catechol	.	.	0.29	.	.
24.	C3-Phenol	.	.	0.03	.	.
25.	C3-Phenol	.	0.12	0.01	.	0.22
26.	Quinoline	.	.	0.04	.	.
27.	C1-Catechol	.	.	0.14	.	0.06
28.	C3-Phenol	.	.	.	.	.
29.	C3-Phenol	.	.	0.01	.	.
30.	2-Methylnaphthalene	0.19	0.18	0.17	0.23	0.34
31.	1-Methylnaphthalene	0.11	0.04	0.15	0.12	0.07
32.	C2-Catechol	.	.	0.02	.	.
33.	Biphenyl	0.22	0.83	0.12	0.30	0.52
34.	C2-Catechol	.	.	0.06	.	0.01
35.	Acenaphthene	0.13	0.12	0.10	0.20	0.13
36.	Dibenzofuran	0.33	0.53	0.18	0.45	0.41
37.	Naphthol	0.26	0.08	0.36	0.39	0.19
38.	Phenanthrene	0.26	0.49	0.16	0.60	0.57
39.	Fluoranthene	0.07	0.14	0.04	0.18	0.10
40.	Pyrene	0.06	0.10	0.03	0.15	0.07
	Total wt%	98.93	98.96	98.23	98.97	98.85
	# of GC Peaks	24	23	37	99	99

KF-Water = Karl Fischer water.

TABLE 4

GC/MS DATA, ADJUSTED, ISOPROPANOL-FREE IN WT%

#	Compound / Sample	237-45	237-46
1.	KF-Water	10.71	15.85
2.	Isopropanol	.	.
3.	Benzene	8.99	23.81
4.	Toluene	3.92	3.01
5.	C2-Benzene	0.28	0.07
6.	mp-Xylene	0.41	0.07
7.	o-Xylene	0.06	.
8.	Aniline	.	0.11
9.	Phenol	42.96	26.77
10.	C3-Benzene	.	.
11.	Indane	0.14	0.06
12.	Indene	0.09	0.06
13.	o-Cresol	1.03	0.10
14.	mp-Cresol	6.22	0.88
15.	Guaiacol	.	.
16.	C2-Phenol	.	.
17.	C2-Phenols	0.05	.
18.	C2-Phenol	0.08	.
19.	C2-Phenol	.	.
20.	C2-Phenol	0.39	.
21.	Naphthalene	1.21	2.23
22.	C1-Guaiacol	.	0.04
23.	Catechol	.	.
24.	C3-Phenol	.	.
25.	C3-Phenol	.	0.09
26.	Quinoline	.	.
27.	C1-Catechol	.	.
28.	C3-Phenol	.	.
29.	C3-Phenol	.	.
30.	2-Methylnaphthalene	0.15	0.14
31.	1-Methylnaphthalene	0.08	0.03
32.	C2-Catechol	.	.
33.	Biphenyl	0.17	0.63
34.	C2-Catechol	.	.
35.	Acenaphthene	0.10	0.09
36.	Dibenzofuran	0.26	0.41
37.	Naphthol	0.20	0.06
38.	Phenanthrene	0.21	0.37
39.	Fluoranthene	0.05	0.11
40.	Pyrene	0.05	0.08
	Total wt%	77.83	75.07
	# of GC Peaks	24	23

Data adjusted to liquid total in HRI letter dated Jan 5, 1988.
KF-Water = Karl Fischer water.

TABLE 5

GC/MS ANALYSIS DATA FOR MHRI5511

#	Compound	Rf	RT(min)	A%	wt%	AR,wt%
1.	KF-Water	.	.	.	.	5.45
2.	Isopropanol	0.53	2.030	.	.	.
3.	Benzene	1.12	5.142	.	.	.
4.	Toluene	1.07	8.441	0.27	0.17	0.16
5.	C2-Benzene	1.02	11.596	0.12	0.08	0.08
6.	mp-Xylene	1.02	11.847	0.19	0.13	0.12
7.	o-Xylene	1.02	12.590	0.12	0.08	0.07
8.	Aniline	0.75	15.486	0.15	0.13	0.13
9.	Phenol	0.56	15.784	39.85	47.12	44.55
10.	C3-Benzene	1.00	16.573	0.06	0.04	0.04
11.	Indane	1.00	16.940	0.04	0.03	0.02
12.	Indene	1.00	17.206	0.08	0.06	0.05
13.	o-Cresol	0.69	17.710	8.05	7.73	7.30
14.	mp-Cresol	0.69	18.355	20.08	19.27	18.22
15.	Guaiacol	0.57	18.607	1.50	1.75	1.65
16.	C2-Phenol	0.75	19.056	0.36	0.32	0.30
17.	C2-Phenols	0.75	20.201	1.60	1.41	1.34
18.	C2-Phenol	0.75	20.238	1.18	1.04	0.98
19.	C2-Phenol	0.75	20.697	1.65	1.45	1.38
20.	C2-Phenol	0.75	20.777	2.83	2.50	2.36
21.	Naphthalene	0.90	21.049	0.38	0.28	0.26
22.	C1-Guaiacol	0.60	21.185	0.08	0.09	0.08
23.	Catechol	0.50	21.547	1.27	1.69	1.59
24.	C3-Phenol	0.86	22.343	0.22	0.17	0.16
25.	C3-Phenol	0.86	22.507	0.25	0.19	0.18
26.	Quinoline	0.56	22.595	0.02	0.03	0.03
27.	C1-Catechol	0.63	23.105	1.77	1.86	1.76
28.	C3-Phenol	0.86	23.226	0.23	0.18	0.17
29.	C3-Phenol	0.86	23.340	0.22	0.17	0.16
30.	2-Methylnaphthalene	1.00	23.919	0.35	0.23	0.22
31.	1-Methylnaphthalene	1.00	24.288	0.39	0.25	0.24
32.	C2-Catechol	0.76	25.305	1.05	0.92	0.87
33.	Biphenyl	0.92	25.903	0.07	0.05	0.05
34.	C2-Catechol	0.76	26.028	1.11	0.97	0.91
35.	Acenaphthene	0.90	28.285	0.05	0.04	0.03
36.	Dibenzofuran	0.90	28.969	0.09	0.07	0.06
37.	Naphthol	0.85	29.036	0.55	0.43	0.41
38.	Phenanthrene	0.94	34.300	0.13	0.09	0.08
39.	Fluoranthene	0.93	39.285	0.02	0.02	0.02
40.	Pyrene	0.93	40.159	0.03	0.02	0.02
	Total A%, wt%, wt%			86.44	91.02	91.51
	# of Peaks, wt counts			37	151.04	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 6

GC/MS ANALYSIS DATA FOR M237-45

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	•	•	•	•	9.23
2.	Isopropanol	0.53	2.030	28.88	35.08	31.84
3.	Benzene	1.12	5.142	14.86	8.54	7.75
4.	Toluene	1.07	8.441	6.18	3.72	3.38
5.	C2-Benzene	1.02	11.596	0.42	0.27	0.24
6.	mp-Xylene	1.02	11.847	0.62	0.39	0.35
7.	o-Xylene	1.02	12.590	0.09	0.06	0.05
8.	Aniline	0.75	15.486	•	•	•
9.	Phenol	0.56	15.784	35.48	40.79	37.03
10.	C3-Benzene	1.00	16.573	•	•	•
11.	Indane	1.00	16.940	0.20	0.13	0.12
12.	Indene	1.00	17.206	0.14	0.09	0.08
13.	o-Cresol	0.69	17.710	1.05	0.98	0.89
14.	mp-Cresol	0.69	18.355	6.33	5.91	5.36
15.	Guaiacol	0.57	18.607	•	•	•
16.	C2-Phenol	0.75	19.056	•	•	•
17.	C2-Phenols	0.75	20.201	0.06	0.05	0.05
18.	C2-Phenol	0.75	20.238	0.09	0.08	0.07
19.	C2-Phenol	0.75	20.697	•	•	•
20.	C2-Phenol	0.75	20.777	0.43	0.37	0.34
21.	Naphthalene	0.90	21.049	1.61	1.15	1.04
22.	C1-Guaiacol	0.60	21.185	•	•	•
23.	Catechol	0.50	21.547	•	•	•
24.	C3-Phenol	0.86	22.343	•	•	•
25.	C3-Phenol	0.86	22.507	•	•	•
26.	Quinoline	0.56	22.595	•	•	•
27.	C1-Catechol	0.63	23.105	•	•	•
28.	C3-Phenol	0.86	23.226	•	•	•
29.	C3-Phenol	0.86	23.340	•	•	•
30.	2-Methylnaphthalene	1.00	23.919	0.22	0.14	0.13
31.	1-Methylnaphthalene	1.00	24.288	0.12	0.08	0.07
32.	C2-Catechol	0.76	25.305	•	•	•
33.	Biphenyl	0.92	25.903	0.23	0.16	0.15
34.	C2-Catechol	0.76	26.028	•	•	•
35.	Acenaphthene	0.90	28.285	0.13	0.10	0.09
36.	Dibenzofuran	0.90	28.969	0.35	0.25	0.22
37.	Naphthol	0.85	29.036	0.25	0.19	0.17
38.	Phenanthrene	0.94	34.300	0.28	0.19	0.18
39.	Fluoranthene	0.93	39.285	0.07	0.05	0.05
40.	Pyrene	0.93	40.159	0.06	0.04	0.04
	Total A%, wt%, wt%			98.16	98.82	98.93
	# of Peaks, wt counts			24	155.30	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 7

GC/MS ANALYSIS DATA FOR M237-46

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	•	•	•	•	11.98
2.	Isopropanol	0.53	2.030	36.65	47.96	42.22
3.	Benzene	1.12	5.142	33.02	20.45	18.00
4.	Toluene	1.07	8.441	3.99	2.59	2.28
5.	C2-Benzene	1.02	11.596	0.09	0.06	0.05
6.	mp-Xylene	1.02	11.847	0.09	0.06	0.05
7.	o-Xylene	1.02	12.590	•	•	•
8.	Aniline	0.75	15.486	0.11	0.10	0.09
9.	Phenol	0.56	15.784	18.56	22.99	20.24
10.	C3-Benzene	1.00	16.573	•	•	•
11.	Indane	1.00	16.940	0.07	0.05	0.04
12.	Indene	1.00	17.206	0.07	0.05	0.04
13.	o-Cresol	0.69	17.710	0.09	0.09	0.08
14.	mp-Cresol	0.69	18.355	0.75	0.75	0.66
15.	Guaiacol	0.57	18.607	•	•	•
16.	C2-Phenol	0.75	19.056	•	•	•
17.	C2-Phenols	0.75	20.201	•	•	•
18.	C2-Phenol	0.75	20.238	•	•	•
19.	C2-Phenol	0.75	20.697	•	•	•
20.	C2-Phenol	0.75	20.777	•	•	•
21.	Naphthalene	0.90	21.049	2.49	1.92	1.69
22.	C1-Guaiacol	0.60	21.185	0.03	0.03	0.03
23.	Catechol	0.50	21.547	•	•	•
24.	C3-Phenol	0.86	22.343	•	•	•
25.	C3-Phenol	0.86	22.507	0.09	0.07	0.07
26.	Quinoline	0.56	22.595	•	•	•
27.	C1-Catechol	0.63	23.105	•	•	•
28.	C3-Phenol	0.86	23.226	•	•	•
29.	C3-Phenol	0.86	23.340	•	•	•
30.	2-Methylnaphthalene	1.00	23.919	0.17	0.12	0.11
31.	1-Methylnaphthalene	1.00	24.288	0.04	0.03	0.02
32.	C2-Catechol	0.76	25.305	•	•	•
33.	Biphenyl	0.92	25.903	0.72	0.54	0.48
34.	C2-Catechol	0.76	26.028	•	•	•
35.	Acenaphthene	0.90	28.285	0.10	0.08	0.07
36.	Dibenzofuran	0.90	28.969	0.45	0.35	0.31
37.	Naphthol	0.85	29.036	0.06	0.05	0.05
38.	Phenanthrene	0.94	34.300	0.43	0.32	0.28
39.	Fluoranthene	0.93	39.285	0.13	0.09	0.08
40.	Pyrene	0.93	40.159	0.09	0.07	0.06
	Total A%, wt%, wt%			98.30	98.82	98.96
	# of Peaks, wt counts			23	144.19	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 8

GC/MS ANALYSIS DATA FOR M237-50

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	.	.	.	.	13.87
2.	Isopropanol	0.53	2.030	35.69	43.29	37.29
3.	Benzene	1.12	5.142	8.15	4.68	4.03
4.	Toluene	1.07	8.441	8.08	4.86	4.18
5.	C2-Benzene	1.02	11.596	0.89	0.56	0.48
6.	mp-Xylene	1.02	11.847	1.17	0.74	0.63
7.	o-Xylene	1.02	12.590	0.25	0.16	0.14
8.	Aniline	0.75	15.486	0.23	0.19	0.17
9.	Phenol	0.56	15.784	22.42	25.74	22.17
10.	C3-Benzene	1.00	16.573	0.01	0.01	0.01
11.	Indane	1.00	16.940	0.32	0.21	0.18
12.	Indene	1.00	17.206	0.18	0.11	0.10
13.	o-Cresol	0.69	17.710	2.50	2.33	2.01
14.	mp-Cresol	0.69	18.355	11.81	11.00	9.48
15.	Guaiacol	0.57	18.607	.	.	.
16.	C2-Phenol	0.75	19.056	0.08	0.07	0.06
17.	C2-Phenols	0.75	20.201	0.26	0.23	0.19
18.	C2-Phenol	0.75	20.238	0.32	0.28	0.24
19.	C2-Phenol	0.75	20.697	0.26	0.22	0.19
20.	C2-Phenol	0.75	20.777	1.13	0.97	0.83
21.	Naphthalene	0.90	21.049	1.27	0.91	0.78
22.	Cl-Guaiacol	0.60	21.185	0.02	0.02	0.02
23.	Catechol	0.50	21.547	0.16	0.21	0.18
24.	C3-Phenol	0.86	22.343	0.03	0.02	0.02
25.	C3-Phenol	0.86	22.507	0.01	0.01	0.01
26.	Quinoline	0.56	22.595	0.02	0.03	0.02
27.	Cl-Catechol	0.63	23.105	0.10	0.10	0.08
28.	C3-Phenol	0.86	23.226	.	.	.
29.	C3-Phenol	0.86	23.340	0.01	0.01	0.01
30.	2-Methylnaphthalene	1.00	23.919	0.19	0.12	0.11
31.	1-Methylnaphthalene	1.00	24.288	0.17	0.11	0.09
32.	C2-Catechol	0.76	25.305	0.02	0.01	0.01
33.	Biphenyl	0.92	25.903	0.12	0.08	0.07
34.	C2-Catechol	0.76	26.028	0.05	0.05	0.04
35.	Acenaphthene	0.90	28.285	0.10	0.07	0.06
36.	Dibenzofuran	0.90	28.969	0.18	0.13	0.11
37.	Naphthol	0.85	29.036	0.35	0.26	0.23
38.	Phenanthrene	0.94	34.300	0.17	0.11	0.10
39.	Fluoranthene	0.93	39.285	0.04	0.03	0.02
40.	Pyrene	0.93	40.159	0.03	0.02	0.02
	Total A%, wt%, wt%			96.81	97.95	98.23
	# of Peaks, wt counts			37	155.56	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 9

GC/MS ANALYSIS DATA FOR M237-51

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	•	•	•	•	13.37
2.	Isopropanol	0.53	2.030	42.43	51.84	44.91
3.	Benzene	1.12	5.142	16.09	9.30	8.06
4.	Toluene	1.07	8.441	7.62	4.61	3.99
5.	C2-Benzene	1.02	11.596	0.41	0.26	0.23
6.	mp-Xylene	1.02	11.847	0.52	0.33	0.29
7.	o-Xylene	1.02	12.590	0.08	0.05	0.04
8.	Aniline	0.75	15.486	•	•	•
9.	Phenol	0.56	15.784	20.65	23.88	20.69
10.	C3-Benzene	1.00	16.573	•	•	•
11.	Indane	1.00	16.940	0.22	0.14	0.12
12.	Indene	1.00	17.206	0.14	0.09	0.08
13.	o-Cresol	0.69	17.710	0.58	0.54	0.47
14.	mp-Cresol	0.69	18.355	4.54	4.26	3.69
15.	Guaiacol	0.57	18.607	•	•	•
16.	C2-Phenol	0.75	19.056	•	•	•
17.	C2-Phenols	0.75	20.201	•	•	•
18.	C2-Phenol	0.75	20.238	•	•	•
19.	C2-Phenol	0.75	20.697	•	•	•
20.	C2-Phenol	0.75	20.777	0.21	0.18	0.16
21.	Naphthalene	0.90	21.049	2.31	1.66	1.44
22.	Cl-Guaiacol	0.60	21.185	•	•	•
23.	Catechol	0.50	21.547	•	•	•
24.	C3-Phenol	0.86	22.343	•	•	•
25.	C3-Phenol	0.86	22.507	•	•	•
26.	Quinoline	0.56	22.595	•	•	•
27.	Cl-Catechol	0.63	23.105	•	•	•
28.	C3-Phenol	0.86	23.226	•	•	•
29.	C3-Phenol	0.86	23.340	•	•	•
30.	2-Methylnaphthalene	1.00	23.919	0.22	0.14	0.12
31.	1-Methylnaphthalene	1.00	24.288	0.12	0.08	0.07
32.	C2-Catechol	0.76	25.305	•	•	•
33.	Biphenyl	0.92	25.903	0.27	0.19	0.16
34.	C2-Catechol	0.76	26.028	•	•	•
35.	Acenaphthene	0.90	28.285	0.17	0.12	0.11
36.	Dibenzofuran	0.90	28.969	0.39	0.28	0.24
37.	Naphthol	0.85	29.036	0.33	0.25	0.22
38.	Phenanthrene	0.94	34.300	0.55	0.38	0.33
39.	Fluoranthene	0.93	39.285	0.16	0.11	0.10
40.	Pyrene	0.93	40.159	0.14	0.09	0.08
	Total A%, wt%, wt%			98.17	98.81	98.97
	# of Peaks, wt counts			22	154.44	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 10

GC/MS ANALYSIS DATA FOR M237-61

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	•	•	•	•	10.51
2.	Isopropanol	0.53	2.030	43.27	55.56	49.72
3.	Benzene	1.12	5.142	24.92	15.14	13.55
4.	Toluene	1.07	8.441	8.79	5.59	5.00
5.	C2-Benzene	1.02	11.596	0.23	0.16	0.14
6.	mp-Xylene	1.02	11.847	0.26	0.18	0.16
7.	o-Xylene	1.02	12.590	0.04	0.03	0.02
8.	Aniline	0.75	15.486	0.17	0.15	0.13
9.	Phenol	0.56	15.784	12.89	15.66	14.02
10.	C3-Benzene	1.00	16.573	•	•	•
11.	Indane	1.00	16.940	0.11	0.08	0.07
12.	Indene	1.00	17.206	0.07	0.05	0.04
13.	o-Cresol	0.69	17.710	0.19	0.19	0.17
14.	mp-Cresol	0.69	18.355	2.20	2.17	1.94
15.	Guaiacol	0.57	18.607	•	•	•
16.	C2-Phenol	0.75	19.056	•	•	•
17.	C2-Phenols	0.75	20.201	0.01	0.01	0.01
18.	C2-Phenol	0.75	20.238	0.02	0.02	0.02
19.	C2-Phenol	0.75	20.697	0.10	0.09	0.08
20.	C2-Phenol	0.75	20.777	•	•	•
21.	Naphthalene	0.90	21.049	2.80	2.12	1.90
22.	Cl-Guaiacol	0.60	21.185	0.04	0.04	0.04
23.	Catechol	0.50	21.547	•	•	•
24.	C3-Phenol	0.86	22.343	•	•	•
25.	C3-Phenol	0.86	22.507	0.15	0.12	0.11
26.	Quinoline	0.56	22.595	•	•	•
27.	Cl-Catechol	0.63	23.105	0.03	0.03	0.03
28.	C3-Phenol	0.86	23.226	•	•	•
29.	C3-Phenol	0.86	23.340	•	•	•
30.	2-Methylnaphthalene	1.00	23.919	0.28	0.19	0.17
31.	1-Methylnaphthalene	1.00	24.288	0.05	0.04	0.03
32.	C2-Catechol	0.76	25.305	•	•	•
33.	Biphenyl	0.92	25.903	0.39	0.29	0.26
34.	C2-Catechol	0.76	26.028	0.01	0.01	0.01
35.	Acenaphthene	0.90	28.285	0.10	0.07	0.06
36.	Dibenzofuran	0.90	28.969	0.30	0.23	0.20
37.	Naphthol	0.85	29.036	0.13	0.11	0.09
38.	Phenanthrene	0.94	34.300	0.44	0.32	0.28
39.	Fluoranthene	0.93	39.285	0.08	0.06	0.05
40.	Pyrene	0.93	40.159	0.05	0.04	0.04
	Total A%, wt%, wt%			98.12	98.72	98.85
	# of Peaks, wt counts			29	146.94	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 11

GC/MS ANALYSIS DATA FOR M237-61RC

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	•	•	•	•	0.17
2.	Isopropanol	0.53	2.030	•	•	•
3.	Benzene	1.12	5.142	0.03	0.03	0.03
4.	Toluene	1.07	8.441	44.48	39.29	39.22
5.	C2-Benzene	1.02	11.596	1.83	1.69	1.69
6.	mp-Xylene	1.02	11.847	2.40	2.23	2.22
7.	o-Xylene	1.02	12.590	0.15	0.14	0.14
8.	Aniline	0.75	15.486	0.17	0.21	0.21
9.	Phenol	0.56	15.784	1.27	2.15	2.14
10.	C3-Benzene	1.00	16.573	•	•	•
11.	Indane	1.00	16.940	•	•	•
12.	Indene	1.00	17.206	•	•	•
13.	o-Cresol	0.69	17.710	0.30	0.41	0.41
14.	mp-Cresol	0.69	18.355	10.42	14.28	14.25
15.	Guaiacol	0.57	18.607	•	•	•
16.	C2-Phenol	0.75	19.056	•	•	•
17.	C2-Phenols	0.75	20.201	0.10	0.13	0.13
18.	C2-Phenol	0.75	20.238	•	•	•
19.	C2-Phenol	0.75	20.697	0.88	1.10	1.10
20.	C2-Phenol	0.75	20.777	•	•	•
21.	Naphthalene	0.90	21.049	6.88	7.23	7.22
22.	C1-Guaiacol	0.60	21.185	0.14	0.22	0.22
23.	Catechol	0.50	21.547	0.05	0.10	0.10
24.	C3-Phenol	0.86	22.343	0.01	0.01	0.01
25.	C3-Phenol	0.86	22.507	•	•	•
26.	Quinoline	0.56	22.595	0.89	1.50	1.49
27.	C1-Catechol	0.63	23.105	0.05	0.07	0.07
28.	C3-Phenol	0.86	23.226	0.18	0.19	0.19
29.	C3-Phenol	0.86	23.340	•	•	•
30.	2-Methylnaphthalene	1.00	23.919	1.51	1.43	1.43
31.	1-Methylnaphthalene	1.00	24.288	0.47	0.45	0.45
32.	C2-Catechol	0.76	25.305	0.02	0.03	0.03
33.	Biphenyl	0.92	25.903	2.15	2.21	2.21
34.	C2-Catechol	0.76	26.028	0.02	0.02	0.02
35.	Acenaphthene	0.90	28.285	0.90	0.94	0.94
36.	Dibenzofuran	0.90	28.969	1.19	1.25	1.25
37.	Naphthol	0.85	29.036	1.32	1.46	1.46
38.	Phenanthrene	0.94	34.300	2.74	2.75	2.75
39.	Fluoranthene	0.93	39.285	0.68	0.69	0.69
40.	Pyrene	0.93	40.159	0.52	0.53	0.53
	Total A%, wt%, wt%			81.74	82.74	82.77
	# of Peaks, wt counts			29	105.79	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

TABLE 12

GC/MS ANALYSIS DATA FOR ML-731

#	Compound	Rf	Rt(min)	A%	wt%	AR,wt%
1.	KF-Water	.	.	.	.	0.09
2.	Isopropanol	0.53	2.030	.	.	.
3.	Benzene	1.12	5.142	.	.	.
4.	Toluene	1.07	8.441	.	.	.
5.	C2-Benzene	1.02	11.596	.	.	.
6.	mp-Xylene	1.02	11.847	.	.	.
7.	o-Xylene	1.02	12.590	0.01	0.01	0.01
8.	Aniline	0.75	15.486	0.30	0.28	0.28
9.	Phenol	0.56	15.784	20.18	25.40	25.38
10.	C3-Benzene	1.00	16.573	0.06	0.05	0.05
11.	Indane	1.00	16.940	.	.	.
12.	Indene	1.00	17.206	.	.	.
13.	o-Cresol	0.69	17.710	7.71	7.88	7.87
14.	mp-Cresol	0.69	18.355	28.52	29.13	29.11
15.	Guaiacol	0.57	18.607	2.13	2.63	2.63
16.	C2-Phenol	0.75	19.056	0.47	0.44	0.44
17.	C2-Phenols	0.75	20.201	0.01	0.01	0.01
18.	C2-Phenol	0.75	20.238	2.58	2.42	2.42
19.	C2-Phenol	0.75	20.697	2.38	2.24	2.24
20.	C2-Phenol	0.75	20.777	4.06	3.82	3.82
21.	Naphthalene	0.90	21.049	0.48	0.38	0.38
22.	Cl-Guaiacol	0.60	21.185	0.11	0.13	0.13
23.	Catechol	0.50	21.547	1.89	2.66	2.66
24.	C3-Phenol	0.86	22.343	0.25	0.20	0.20
25.	C3-Phenol	0.86	22.507	0.36	0.29	0.29
26.	Quinoline	0.56	22.595	0.05	0.07	0.07
27.	Cl-Catechol	0.63	23.105	2.47	2.76	2.76
28.	C3-Phenol	0.86	23.226	0.24	0.19	0.19
29.	C3-Phenol	0.86	23.340	0.22	0.18	0.18
30.	2-Methylnaphthalene	1.00	23.919	0.28	0.20	0.20
31.	1-Methylnaphthalene	1.00	24.288	0.56	0.39	0.39
32.	C2-Catechol	0.76	25.305	1.45	1.35	1.35
33.	Biphenyl	0.92	25.903	0.09	0.07	0.07
34.	C2-Catechol	0.76	26.028	1.74	1.62	1.62
35.	Acenaphthene	0.90	28.285	0.07	0.05	0.05
36.	Dibenzofuran	0.90	28.969	0.13	0.10	0.10
37.	Naphthol	0.85	29.036	0.73	0.61	0.61
38.	Phenanthrene	0.94	34.300	0.15	0.11	0.11
39.	Fluoranthene	0.93	39.285	0.03	0.02	0.02
40.	Pyrene	0.93	40.159	0.04	0.03	0.03
	Total A%, wt%, wt%			79.74	85.72	85.73
	# of Peaks, wt counts			32	141.86	

KF-Water = Karl Fischer water; Rf = FID relative response factor; Rt = GC retention time (min); A% = GC area percent; wt% = weight percent; AR = as received; wt counts = sum of A%/Rf;

APPENDIX B

Energy & Mineral Research Center

Fuels & Process Chemistry Research Institute
ND Mining & Mineral Resources Research Institute
Combustion & Environmental Systems Research Institute

Box 8213, University Station / Grand Forks, North Dakota 58202 / Phone: (701) 777-5000 / Fax: 777-6181

January 13, 1989

Mr. Alfred Kuhn
Dakota Gasification Company
P.O. Box 1149
Beulah, ND 58523

SUBJECT: FINAL REPORT

Dear Fred:

Enclosed is the final report that summarizes the work on the analysis of the rectisol naphtha stream. You have the detailed data for the analyses. Please let me know if additional data is desired.

Highlights include the azeotropic distillation of benzene which results in no clear distillation temperature for this compound (probably due to the presence of ketones) and that heavier sulfur compounds are present which decompose to small odoriferous sulfur compounds during distillation (the last fractions are the most odiferous). Also, about 77% of the total sulfur was present in the end-pot fraction.

Thank you for the opportunity of providing this research for DGC.

Sincerely,

Curtis L. Knudson

Curtis L. Knudson
Manager, Process Chemistry

CLK/napfin

Enclosure

SCHOOL OF ENGINEERING & MINES
University Of North Dakota

FINAL REPORT

RECTISOL NAPHTHA ANALYSIS

FOR

DAKOTA GASIFICATION COMPANY

Contract No. 88-PMM-059

A. Kuhn

PREPARED BY

Curtis L. Knudson

University of North Dakota

January 13, 1989

Executive Summary:

Rectisol naphtha was comprehensively analyzed to determine distillation characteristics as well as compounds present, and their concentrations. Highlights of the research includes the azeotropic distillation of benzene which results in no clear distillation temperature for this compound (probably due to the presence of ketones) and that heavier sulfur compounds are present which decompose to small odoriferous sulfur compounds during distillation (the last distillate fractions are the most odoriferous). Also, about 77% of the total sulfur was present in the end-pot fraction. The presence of basic nitrogen compounds was established.

Summary of the Revised Naphtha Work Plan:

The work plan was revised to reflect a budget of $27,000 rather than $50,000 in the original contract. Data reduction and preparation time was decreased. Data not reduced was provided directly to DGC for their evaluation. The following work plan was followed:

1. Naphtha TBP distillation to 7 cuts.

2. Analysis of the Total sample and each distillation cut.

    a. Water, CHN, and Gravity
    b. GC/FTIR/FID of Total sample only.
    c. GC/FID
    d. GC/MS with identities of nitrogen, sulfur, and oxygenated species, as in original proposal.
    e. Proton NMR
    f. IR
    g. UV
    h. Metal (potential catalyst poisons)

3. Reporting

    a. Reports as specified by the client to the extent of funding to include:
        - letter monthly reports.
            1st monthly mailed November 14, 1988
            2nd monthly mailed November 29, 1988
        - Final report summary with copies of data.
            mailed January 13, 1989
        - Final Meeting to discuss report and data.
            held December 13, 1988 at Beulah, ND

4. Changes reflected in above to original contract.

   a. A more limited determination of trace organics.
   b. No oxygen by direct determination.
   c. Much less interpretation and written discussion of the data.
   d. In general, a necessity for a "first try" success in analysis. Analysis problems will be documented and result in a decrease in the number of tests performed.

Results:

1. Naphtha TBP distillation (12-plate column) to 7 cuts:

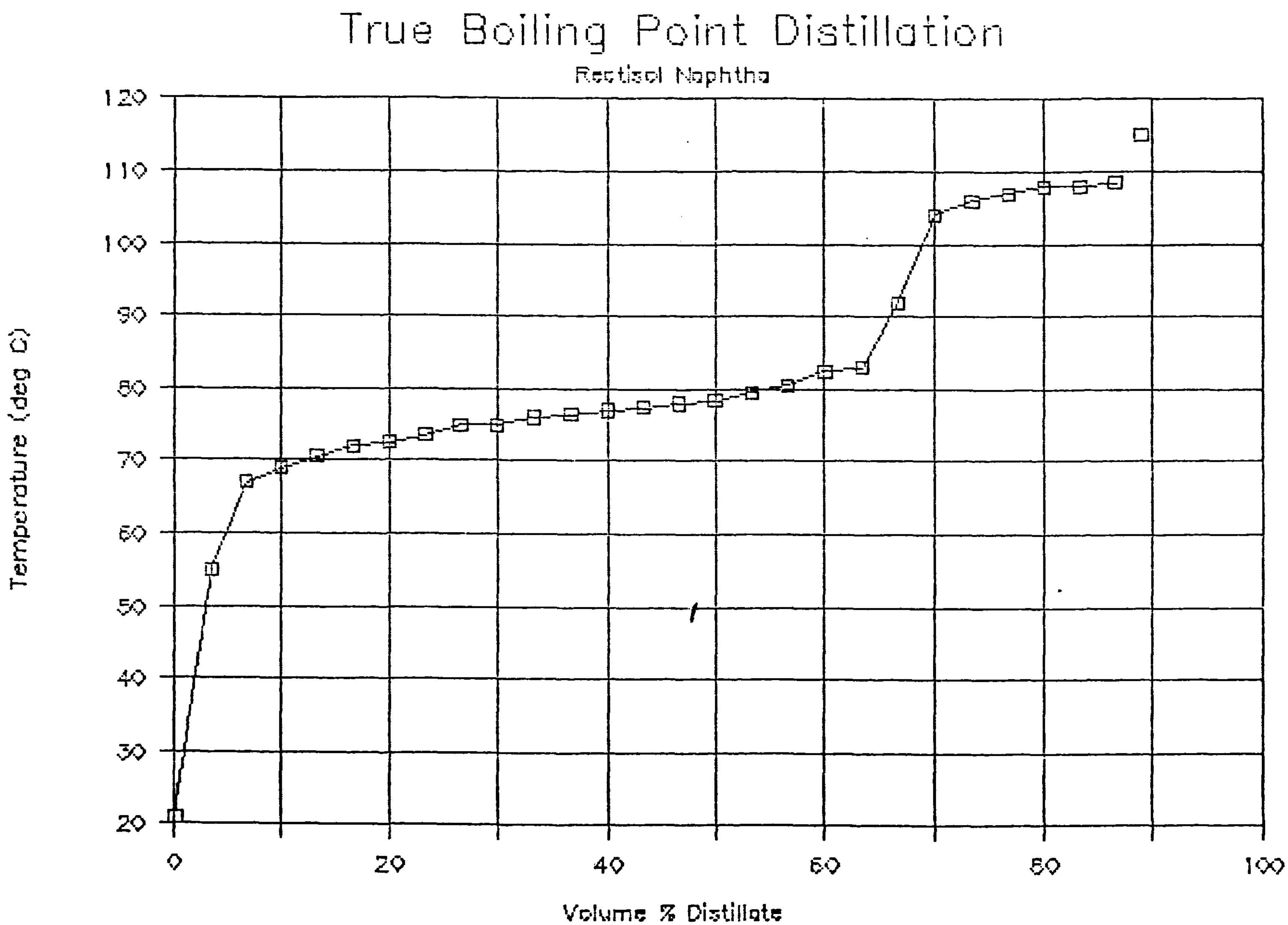
The distillation curve (Figure 1) shows the presence of inflection points between 60 and 85° potentially due to azeotropic combinations. Seven fractions were collected for analysis. The distillation data for the fractions analyzed are reported on Table 1 (the appendix of the monthly contains data on the first distillation). A recovery of 98.1 wt% was achieved using a cooled system to prevent evaporative losses. Normalized data was used in subsequent calculations. A copy of the Lotus spreadsheet and calculations can be obtained upon request.

2. Analysis of the total sample and each distillation cut:

   a. Water, CHN, S, and Gravity:

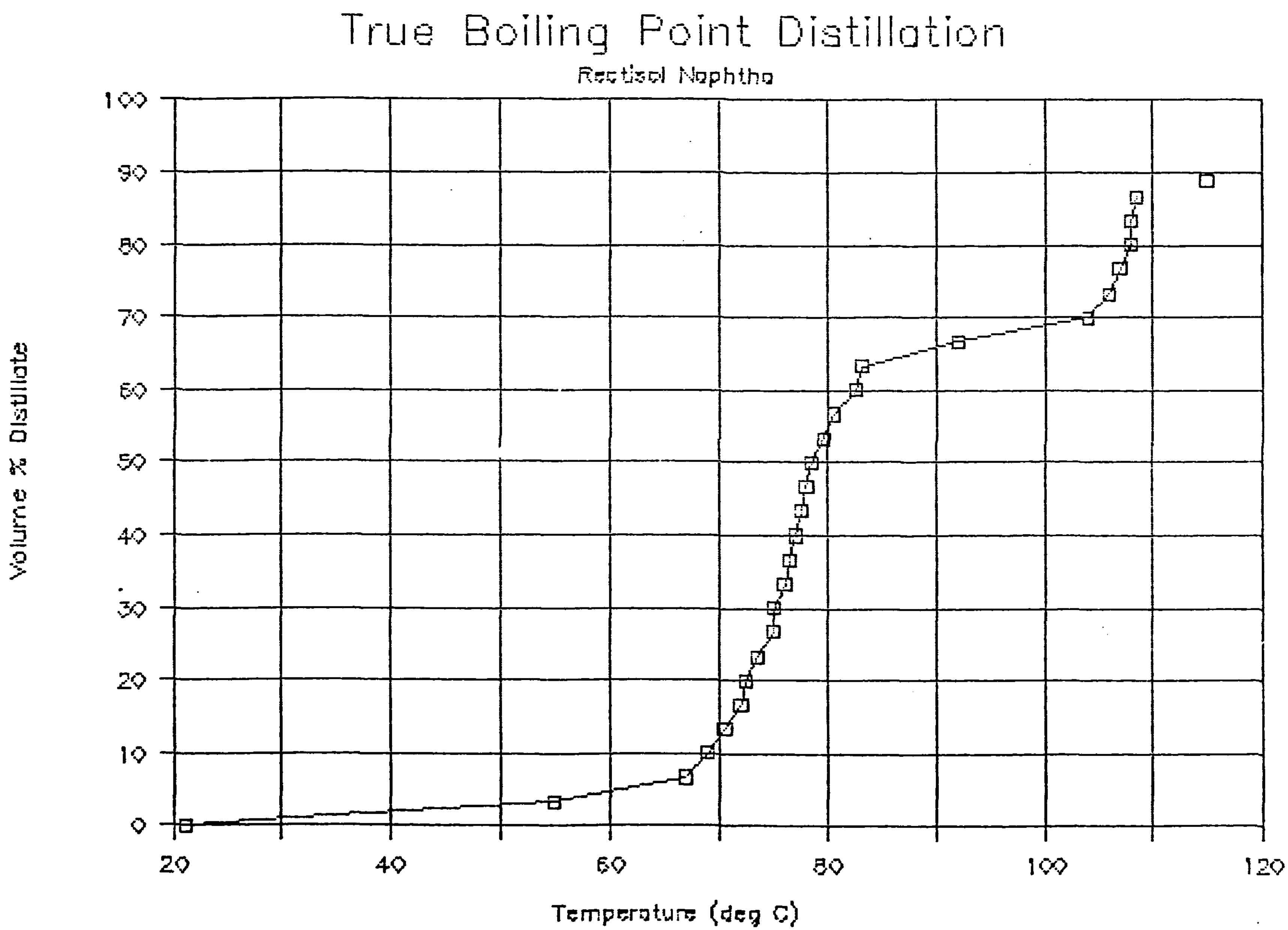
Acetone, methanol, and benzene known samples were analyzed in the same time frame as the naphtha samples to verify C, H, and O values. Mixtures of pyridine in toluene were used to calibrate for nitrogen. Calculated carbon values were 0.1 to 0.7% higher than the measured values (62.00 versus 62.07 wt% for acetone), hydrogen values were 0.9 to 2.7% lower (10.49 versus 10.34 for acetone), and oxygen values calculated by difference were 0.2% higher (27.51 versus 27.59). The initial analysis of the naphtha sample indicated a nitrogen value of zero. Standards were made up containing various amounts of pyridine in toluene and analyzed. Re-analysis of the naphtha indicated a nitrogen value of 0.14 plus or minus 0.08. In subsequent calculations the nitrogen value in naphtha has been assumed to be zero, since all distillation fractions also gave values of zero. Complete data was supplied to DGC. Average calibration errors were plus or minus 0.07, 0.02, and 0.09 for C, H, and N, respectively.

Mass-balanced data is presented in Table 2 while the data appendix supplied to DGC contained detailed calculation information. Distillation wt% values and water values were normalized to 100% and to the total sample measured water value, respectively. The elemental data for the fractions are reported



True Boiling Point Distillation
Rectisol Naphtha
Temperature (deg C)
120
110
100
90
80
70
60
50
40
30
20
0
20
40
60
80
100
Volume % Distillate

Figure 1a.



True Boiling Point Distillation
Rectisol Naphtha
Volume % Distillate
Temperature (deg C)
0
10
20
30
40
50
60
70
80
90
100
20
40
60
80
100
120

Figure 1b.

TABLE 1

True Boiling Point Distillation 3

Rectisol Naphtha

Sp. Gravity	0.831	Room Temp	21 deg C
Sample wt.	249.3 g	Baro Press	738.2 mm Hg
Sample Vol.	300 ml	Condenser Temp	0 deg C

Distillation Data

	ml Dist	Vol %	Temp(C)	Time(min)			Wt %	Density
IBP	0	0.0	21	25:20				
	10	3.3	55	36:00				
	20	6.7	67	42:00				
	30	10.0	69	47:00				
	40	13.3	70.5	52:00	Fr 1	45ml	14.1	0.782
	50	16.7	72	57:00				
	60	20.0	72.5	62:00				
	70	23.3	73.5	67:40	Fr 2	35ml	11.6	0.826
	80	26.7	75	73:40				
	90	30.0	75	78:20				
	100	33.3	76	84:20				
	110	36.7	76.5	90:30				
	120	40.0	77	96:50				
	130	43.3	77.5	103:20				
	140	46.7	78	109:40				
	150	50.0	78.5	115:50	Fr 3	75ml	25.5	0.848
	160	53.3	79.5	119:50				
	170	56.7	80.5	124:00				
	180	60.0	82.5	130:50				
	190	63.3	83	139:40	Fr 4	37ml	12.4	0.835
	200	66.7	92	146:20				
	210	70.0	104	153:40				
	220	73.3	106		Fr 5	28ml	9.3	0.832
	230	76.7	107	163:30				
	240	80.0	108	168:20				
	250	83.3	108	173:30				
	260	86.7	108.5	181:20	Fr 6	47ml	15.8	0.838
	270	90.0						
	280	93.3						
	290	96.7						
	300	100.0						
Max Temp			115	199:00				
Max Vol		89 %	267 ml					
Pot Resid.						27.3	9.3	0.853
Recovery							98.1	

TABLE 2

RECTISOL NAPHTHA DISTILLATION AND ELEMENTAL DATA

	Feed Amount		Distillate Fraction F-1	F-2	F-3	F-4	F-5	F-6	F-7	Recovery	Error wt%
Sample wt	100.00		14.10	11.60	25.50	12.40	9.30	15.80	9.30	98.00	2.00
		Norm.	14.39	11.84	26.02	12.65	9.49	16.12	9.49	100.00	
meas.%H2O	0.27		0.50	0.09	0.09	0.06	0.07	0.18	0.82	85.19	0.04
As Recieved											
%H2O	0.27		0.59	0.11	0.11	0.07	0.08	0.21	0.97	100.01	-0.005
C	86.76		81.17	86.63	89.28	89.60	88.60	88.78	79.48	100.00	0.000
H	9.75		10.72	9.77	9.19	9.23	9.90	9.78	10.28	0.00	0.001
N	0.00									99.77	0.005
diff O	2.52		7.76	3.46	1.41	1.09	1.25	0.80	2.48	100.16	-0.002
S	0.97		0.35	0.14	0.12	0.08	0.25	0.64	7.76	100.00	0.000
Ash	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00	100.00	0.000
TOTAL	100.00		100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	0.000
Moisture Free											
%C	86.99		81.66	86.72	89.38	89.66	88.67	88.97	80.26		
H	9.75		10.72	9.77	9.19	9.23	9.90	9.78	10.27		
N	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00		
diff O	2.29		7.27	3.37	1.32	1.03	1.18	0.61	1.63		
S	0.97		0.35	0.14	0.12	0.08	0.25	0.64	7.84		
TOTAL	100.00		100.00	100.00	100.00	100.00	100.00	100.00	100.00		
Gravity	0.8363		0.8158	0.8453	0.8408	0.8422	0.8949	0.8446	0.8885		

File: Naphtha3

as measured. Only the addition of 0.02 wt% units to carbon and the subtraction of 0.09 units of hydrogen from the total sample values were required to close carbon and hydrogen values with that in the sum on the fractions. The sulfur value for naphtha was 1.31 wt% or 0.34 units high after its first analysis. Upon reanalysis a value of 1.01 wt% was obtained, and it required the subtraction of only 0.03 units to obtain a mass balance. Nitrogen contents were below the detection level except as noted previously in the nitrogen calibration test. Small quantities of pyridines have been extracted with acid and quantified by GC/MS analysis.

Gravity data for the total sample and the fractions is presented in the last line of Table 1.

b. GC/FTIR/FID:, c. GC/FID:, and d. GC/MS Results:

GC/FID triplicated or duplicated data on the total sample and each fraction were obtained. Averaged data, wt% data, and a compound mass balance were calculated using computer programs, and identities were determined using GC/FTIR, GC/MS, and retention time information.

Over 300 compounds have been observed in the rectisol naphtha sample received from DGC. However, twenty compounds comprise 75 wt% of the total sample. The balance is composed of numerous cyclopenta- and cyclohexa- olefins in small concentrations and higher boiling cyclopentadiene (CDP) dimers and other material that decomposed to small sulfur-containing compounds and cycloolefins. Nitrogen base compounds were present in small concentrations.

Benzene and alkylated benzenes are major constituents in naphtha as seen in Table 3. Benzene was present in fractions 1-5 and toluene in fractions 5 and 6. The C2- and C3-benzenes were present in the end pot (fraction 7) which made up 9.3 wt% of the total sample. The nature of the distillation profile indicated that benzene could not be separated by simple distillation from the lower boiling components (acetone, etc.) since azeotropes seemed to be present. Benzene could be separated from xylenes and higher boiling components in fractions 6 and 7. However, since numerous cyclopentadienes distill with benzene, CPD dimers would reform since they exist in equilibrium with the monomers.

TABLE 3

BENZENES IN NAPHTHA

Benzene	43.7	wt%
Toluene	17.1	
C2-Benzene isomers	1.04	
	2.51	
	0.08	
	0.71	
C3-Benzene isomers	0.04	
	0.03	
	0.11	
	0.04	
	0.10	
	0.05	
Total	65.5	wt%

Oxygenated compounds are also present in significant quantities as seen in Table 4. Most are ketones and are present in all distillation fractions. They account for 89% of the oxygen in naphtha. Ketones do have a high solubility in water and extraction at the proper temperature and salt contents may remove them from the naphtha.

TABLE 4

OXYGENATED COMPOUNDS IN NAPHTHA

Acetaldehyde	0.20	wt%
Acetone	3.96	
2-Butanone	4.52	
2-Pentanone	0.49	
Total	9.2	wt%

Sulfur compounds are also present (see Table 5) and contaminate all distillate fractions. Methanethiol and the thiiarenes are unique. Methanethiol is very low-boiling and was observed in the analysis of the total sample. However, it was also observed in increasing amounts in fractions 5, 6, and 7. The low-boiling thiiarenes were not observed in the total sample but are observed in the latter distillation fractions. Four light cycloolefins also appear simultaneously in these same fractions in increasing amounts. Mass spectral data confirms the presence of large compounds (CPD dimers) in the end pot that could fragment to give masses corresponding to those given by these light thiiarenes and cycloolefins. It appears that higher boiling compounds containing sulfur decompose during the distillation (or in the GC detector) to lighter material. The end pot also contained a sizable amount of sulfur (77% of the total sulfur in the naphtha) in difficult to identify compounds. Disulfides or elemental sulfur may be present.

TABLE 5

SULFUR COMPOUNDS IN NAPHTHA

	Total	Sum
Methanethiol	0.22 wt%	0.17 wt% **
Thiiarene	. *	0.08 **
C1-thiiarene	.	0.07 **
Thiophene	0.36	0.64
2-Methylthiophene	0.21	0.43
3-Methylthiophene	0.09	0.12
Ethylthiophene	0.07	0.07
C2-Thiophene isomers	.	0.02
	0.02	0.04
Total of compounds	1.0	1.7 wt%

* Below integrator detection level.
** Only present in distillate fractions 6, 7, and 8 in increasing amounts.

No nitrogen compounds were directly observed in the total sample (except during calibration) or the fractions. The elemental nitrogen value of essentially zero was verified by the analysis of known mixtures, which indicated a detection level of 0.02 wt% but a potential error of 0.09 wt%. To determine semi-quantitatively the amount and type of nitrogen compounds present in naphtha, the sample was extracted with acid and the acidic solution was basified and extracted with methylene chloride. The resulting solution was spiked with a known amount of deuterated pyridine, and analyzed by GC/MS. Table 6 presents a list of the compounds identified and concentrations in ppm of pyridine. However, the data may have a high error (up to a factor of four). The decomposition of a compound like pyridine methylsulfide could help account for the appearance of methanethiol.

TABLE 6

NITROGEN COMPOUNDS IN NAPHTHA

Pyridine	27.5	ppm
2-Methylpyridine	8.6	
3 or 4-Methylpyridine	0.8	
C2-Pyridine isomers	1.7	
	0.4	
C3-Pyridine isomers	0.1	
	5.2	
C4-Pyridine isomers	0.3	
	0.3	
	0.7	
Methylpyridine sulfide	0.03	
Unknown (m/e 60,71,103,163)	3.5	
	0.2	
	0.2	
Total	50	ppm

e. Proton NMR:

Proton NMR data were obtained for the total sample and the fractions as well as detail on the olefin region. Results are summarized as follows:

- Olefins of many types are in each fraction in similar amounts.
- Toluene is not present significantly until fraction 5.
- Higher aromatics, more alkylated aromatics, and aliphatics are present in fraction 7 (the end pot).

The NMR spectra were given to DGC for any further evaluation.

f. IR:

IR spectra were obtained neat for the total sample and the fractions. Since the samples were run "neat" results could be calculated to absorbance and quantified. A rich amount of carbonyl and sulfur functionalities are present. The IR spectra were given to DGC for their evaluation.

g. UV:

A limited number of UV spectra were obtained. Data reduction to fourth derivatives, etc. would be required to obtain useful information. The UV spectra were given to DGC for their evaluation.

h. Metal (potential catalyst poisons):

The trace element analysis of rectisol naphtha was performed using atomic spectroscopy. An initial screening was performed using inductively coupled argon plasma spectrometry (ICAP). Quantitative analyses of select elements were performed using ICAP, furnace atomic absorption (AA), and flame (AA).

The original sample was divided into portions. One of these portions was evaporated using gentle heating (<60 degrees Celsius) and another was maintained for analysis of the neat liquid. Each of these portions was digested by reacting with sulfuric acid followed by treatment of the resultant char with nitric acid. The following four solutions were used in the analyses for trace elements:

1. Neat rectisol naphtha.
2. Concentrated naphtha by a factor of 13.4
3. Acid digest of neat naphtha.
4. Acid digest of concentrated naphtha.

The two acid digests added an additional dilution factor of approximately 50 but due to the relative ease of analyzing aqueous samples these yielded the most valuable data.

Table 7 shows the elements chosen and levels detected during the initial ICAP screening test. Some of the elements, although reported as below the detection limit, were selected for more accurate low level quantitation using furnace AA, flame AA, or ICAP as appropriate. The basis of the selection process was the presence of a discernible peak on the scan of the emission which was done for all of the elements. Results of the quantitative analysis of select elements is shown in Table 8. All elements with the exception of iron were determined on the aqueous digest of concentrated rectisol naphtha. Iron was determined on neat naphtha using furnace AA with Zeeman background correction and a xylene solution of tris(1-phenyl-1,3-butanediono) iron (III). A value of 30 ppb was obtained using external standard calibration as well as standard addition methods, confirming the accuracy of the analysis.

TABLE 7

SCREENING OF RECTISOL NAPHTHA USING ICAP

	ELEMENT	CONC. (ppm)
1	Al	< 1
2	As	< 3
3	B	< 3
4	Be	< 0.3
5	Bi	< 0.3
6	Ca	< 3
7	Cd	< 0.3
8	Cr	< 1
9	Cu	< 3
10	Fe	< 1
11	Ga	< 3
12	Ge	< 1
13	Hg	< 3
14	K	< 3
15	Mg	< 3
16	Mn	< 0.1
17	Mo	< 0.3
18	Na	< 29
19	Ni	< 1
20	P	< 3
21	Pb	< 3
22	Sb	< 7
23	Se	< 7
24	Sn	< 6
25	Ti	< 1
26	Tl	< 15
27	V	< 1
28	Zn	< 0.3
29	Zr	< 0.3

TABLE 8

CONFIRMATORY QUANTITATION OF SELECT ELEMENTS

ELEMENT	CONCENTRATION
ANTIMONY	< 14 ppb (1)
IRON	30 ppb (1)
SELENIUM	4.8 ppm (1)
SODIUM	< 4 ppm (2)
TIN	< 1.5 ppm (3)

(1) FURNACE AA - ZEEMAN BACKGROUND CORRECTION
(2) FLAME AA
(3) ICAP

*U.S.Government Printing Office:1990—748-056/24393